

***Final Sampling and Analysis Plan
Volume II: Quality Assurance
Project Plan***

***Waukegan Manufactured Gas
and Coke Plant Site
Waukegan, Illinois***

Prepared for North Shore Gas Company

***Under the Administrative Order on Consent Re: Remedial Investigation and
Feasibility Study for the Waukegan Manufactured Gas and Coke Plant Site
Waukegan, Illinois***

October 24, 1991

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Barr
Engineering Company
8300 Norman Center Drive
Minneapolis, MN 55437
Phone: (612) 832-2600
Fax: (612) 835-0186

DRAFT SAMPLING AND ANALYSIS PLAN
VOLUME II: QUALITY ASSURANCE PROJECT PLAN
WAUKEGAN MANUFACTURED COKE AND GAS PLANT SITE

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VOLUME II: QUALITY ASSURANCE PROJECT PLAN
WAUKEGAN MANUFACTURED COKE AND GAS PLANT SITE

Prepared by: Barr Engineering Co. Date: October 17, 1991

Approved: _____ Date: _____
Mary Mackey
Barr Engineering Co.
Quality Assurance Officer

Approved: _____ Date: _____
Michael Relf
Barr Engineering Co.
Project Manager

Approved: _____ Date: _____
Patrick Doyle
Vice President
North Shore Gas

Approved: _____ Date: _____
Valerie Jones
U.S. EPA Region V
Quality Assurance Officer

Approved: _____ Date: _____
Cindy Nolan
U.S. EPA Region V
Project Manager

Approved: _____ Date: _____
Mary Wisdom
Quality Assurance Coordinator
CH₂M Hill Laboratory

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<u>Name</u>	<u>Affiliation</u>	<u>No. Copies</u>
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Rick Hersemann	PRC Engineering	2
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Jerry Picha	North Shore Gas	1
Russell Selman	Bell, Boyd, & Lloyd	1
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Mike Relf	Barr Engineering Co.	1
Tally Wright-Wells	Barr Engineering Co.	1
Steve Iverson	Barr Engineering Co.	1
John Fox	Barr Engineering Co.	1
Will Mielke	Barr Engineering Co.	1
Karlene French	Barr Engineering Co.	1

SECTION 3
PROJECT DESCRIPTION

3.1 INTRODUCTION

The United States Environmental Protection Agency (EPA) requires that all EPA contractors participate in a centrally managed quality assurance (QA) program. That requirement applies to all environmental monitoring and measurement efforts mandated or supported by the EPA. Each contractor generating data has the responsibility to implement minimum procedures to determine that the precision, accuracy, completeness, and representativeness of its data are known and documented. To determine that the responsibility is met uniformly, each EPA contractor must prepare a written Quality Assurance Project Plan (QAPP) covering each project it is contracted to perform.

This QAPP presents the organization, objectives, functional activities and specific QA and quality control (QC) activities associated with sampling and analysis activities as part of the work plan to implement the remedial investigation at the Waukegan Manufactured Gas and Coke Plant (WCP) Site.

This section describes the site location, the existing conditions at the site, site history, a summary of available information about the geology and groundwater flow at the site, and the findings of previous investigations at the site. Based on that understanding of the site, target compounds are identified, project objectives are defined, and data quality objectives are developed.

3.1.1 Approach to RI/FS

The field investigation for the RI is to be conducted in two phases. The major objectives of the first phase are: 1) to provide information on the location, nature, and horizontal extent of potential contaminant source areas; and 2) to provide preliminary information on groundwater flow patterns and groundwater quality. These data will be used to focus soil and groundwater investigations in Phase II. Activities to be conducted during each phase of the RI are outlined below.

Phase I activities include test trenching to assess the horizontal extent of soil contamination from known site operations (i.e., manufactured gas plant, coking plant, and creosoting facility operations) using field screening techniques. Field screening techniques are particularly relevant to investigations of manufactured gas/coking plant and creosoting sites because wastes from such facilities generally discolor and leave a distinctive oily residue in materials they encounter. Representative samples of visually contaminated soils will be analyzed to assess soil quality. In addition, surficial soil samples will be collected from outside the areas of known site operations to assess the possible presence of a broad range of chemical parameters. Soil samples will also be collected from predetermined off-site locations to assess background soil quality.

Piezometers and monitoring wells will be installed during Phase I to provide a preliminary assessment of groundwater flow patterns and to evaluate the quality of groundwater flowing off-site.

Phase II of the RI will utilize data from Phase I to focus additional soil and groundwater investigations. Soil borings will be placed in areas of

identified contamination (based on the test trenching program) to evaluate the vertical extent of soil contamination and confirm the horizontal extent determined from Phase I. Analyses of soil samples will be performed to quantify levels of contamination and confirm areas identified as uncontaminated. Within each identified area of contaminated soils, interpretations of the extent of vertical contamination will be required for locations where soil borings are not placed. These interpretations will be based on information from soil borings placed in zones of similar shallow contamination (based on Phase I results) within that area.

The locations of monitoring wells placed in Phase II will be selected on the basis of groundwater flow information (including modeling) and contaminant source area identification developed from Phase I information. Data from Phase II wells will be used to refine groundwater flow and quality assessments. The groundwater quality data will also supplement soil boring data for identifying and characterizing source areas.

3.2 SITE DESCRIPTION

3.2.1 Site Location

The WCP site is located in the City of Waukegan, Waukegan Township, Lake County, Illinois. Based on a legal description from a real estate appraisal (Real Estate Research Corporation, 1971) the site is located in the northwest quarter of Section 22, Township 45 North, Range 12, East of the Third Principal Meridian. The site is bounded on the north by Pershing Road (Sea Horse Drive), on the east by Pershing Road, on the south by OMC Plant No. 1, and on the west by Waukegan Harbor. The harbor is maintained and operated by the Waukegan Port District. The site is more or less rectangular in shape

with a total area of 36.37 acres. The location of the WCP site is shown in Figure 3.2-1.

3.2.2 Existing Site Conditions

The only remaining structure from the former Coke Plant is an office building in the southeast corner of the property and the above-grade tar tank foundation. The office building has been remodeled and is currently used by OMC. The northwest portion of the site is used by Larsen Marine to store boats and boat cradles. A new boat slip has been constructed in the northwest quadrant of the site. The western portion of the site contains dredge spoils from Lake Michigan which were placed at the direction of the Army Corps of Engineers in 1974 for temporary storage. A tower that is used for OMC product testing is located just southeast of the dredge spoils. Near the product testing tower is an above ground storage tank farm. There are nine tanks ranging in volume from 300 to 20,000 gallons which contain gasoline, fuel oil, and kerosene. Public parking in the central area of the site has occurred for special events on the Waukegan public beach. In addition, the foundations of several former plant facilities still exist below grade.

3.2.3 Site Hydrogeologic Setting

Geology -- The uppermost deposits at the site are composed of fill. The fill is approximately two to four feet thick. It varies in composition from fine to coarse, brown to black sand and is mixed with demolition debris (Canonie, 1990).

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The fill is reportedly underlain by a black to brown, fine to coarse, sand unit (Canonie, 1990). Much of this unit at the site may have been disturbed during the harbor construction. It is likely composed of both natural in-place material mixed with other native material that was moved from nearby locations and placed at the site as fill. The unit ranges in thickness up to approximately 10 feet.

Underlying the black to brown sand is gray sand unit composed of fine to medium sand with some silt. This unit is approximately 15 to 25 feet thick. Near the new slip location, the unit apparently extends to the surface and the black to brown sand unit is absent. Lenses of silty sand are also present within the gray sand unit.

Samples from the majority of soil borings placed in the vicinity of the proposed new slip indicate the presence of a 1 to 3-foot thick sand and gravel unit directly underlying the gray sand unit. Test holes completed in 1927 on the City Waterworks property (located approximately 1,000 feet south of the site) also encountered a gravel unit at the base of the gray sand.

Underlying the gravel (or directly underlying the gray sand where the gravel is absent) is a thick till deposit of gray silt and clay. This unit was also described, (in a 1927 test hole located south of the coke ovens) as being blue and containing clay, stones, and pebbles. The till unit is reported to be approximately 50 to 200 feet thick regionally (Lineback, 1979). At a soil boring located near the new slip construction area, the unit was at least 30 feet thick; its base was not encountered. The boring completed as part of a well installation near the boiler room (prior to 1927) encountered the base of the till at an elevation of approximately 490 feet

above mean sea level (MSL). At that location, the till was approximately 40 feet thick.

Underlying the till is a thick sequence of carbonate bedrock units. The only boring deep enough to penetrate into the bedrock was the boring for the Boiler Room well. The top of the "limestone" unit was encountered at an elevation of 490 feet MSL (a depth of 91 feet). The well boring extended 43.8 feet into the limestone and no significant changes in lithology were noted. Regionally, the Racine, Waukesha, Joliet, Kankakee, and Edgewood Formations form the uppermost bedrock unit. Together, these units are reported to be in excess of 500 feet thick.

Groundwater Flow -- Limited hydrogeologic studies have been conducted at the WCP site. A groundwater investigation (JRB, 1981) was conducted in the vicinity of the drainage ditch at the OMC Plant No. 2 located north of the WCP site. Additionally, a groundwater flow model was constructed as part of an assessment of impacts associated with construction of the new slip, and groundwater quality data from two monitoring well nests installed at the WCP site are available.

Regionally, groundwater generally occurs under unconfined conditions in the unconsolidated deposits and groundwater in the upper bedrock aquifer occurs under confined conditions. Lake Michigan acts as a major regional discharge zone for groundwater. Therefore, groundwater flow in both the surficial unconsolidated deposits and bedrock units in the region is typically toward the lake.

OMC Plant No. 2 Site Hydrogeology -- Results of the hydrogeologic investigation completed as part of the OMC technical and witnessing case

support (JRB, 1981) indicated that shallow groundwater at the site generally flowed to the east toward Lake Michigan; however, several other factors also had some effect in controlling the localized groundwater flow pattern. These included: (1) the water level in a drainage ditch north of the OMC Plant No. 2, (2) precipitation events, (3) the presence of the till (silt) below the sandy near-shore lake deposits, and (4) fluctuation of the lake level.

Because the investigation focused on the area north of the OMC Plant No. 2 facility, horizontal hydraulic gradient information is available primarily for flow to and from the drainage ditch. Horizontal hydraulic gradients ranged from approximately 6×10^{-3} feet/foot to the southeast along the western boundary of the OMC Plant No. 2 site, to approximately 8×10^{-3} feet/foot in both a northerly and southerly direction along the drainage ditch. No information about horizontal hydraulic gradients was available for the WCP site which is located south of the OMC Plant No. 2 facility. Vertical hydraulic gradients in the surficial aquifer north of the OMC facility are reported to be in a generally upward direction (JRB, 1981).

Hydraulic gradients between the surficial aquifer and the Silurian bedrock were also reported to be in an upward direction (Canonie, 1989). One of the two piezometers installed into the Silurian bedrock reportedly flowed at the surface. Information on the magnitude of the upward gradient was not included in this report.

"Baildown" tests (slug tests) were conducted in 22 monitoring wells screened in the surficial unconsolidated materials at the OMC Plant No. 2 site. Hydraulic conductivities ranged from 2×10^{-4} cm/sec to 9×10^{-3} cm/sec (JRB, 1981).

WCP Site Hydrogeology -- Geraghty and Miller, Inc. (1990) prepared a single layer groundwater flow model of the WCP site. They used the USGS MODFLOW computer code to calculate hydraulic head distributions across the site and groundwater fluxes to the harbor. No site-specific hydrogeologic data were available for actual groundwater flow conditions at the WCP site. As a result, the model could not be calibrated or validated. Results of the modeling predicted that groundwater flow would be to both Lake Michigan and Waukegan Harbor for the simulated conditions, with the divide located approximately down the center of the peninsula. Computed groundwater flow at the northern and southern boundaries of the site had a more southerly component as compared to computed flow at the center of the peninsula.

3.3 SITE HISTORY AND BACKGROUND

3.3.1 Wood Treating Plant

Based on information obtained from the Elgin, Joliet and Eastern Railway Company (EJ&E, 1990), the first industrial facility located on the site was a wood treating plant. This operation was located on the western portion of the site, and was operated by the Chicago Tie and Timber Company from approximately 1908 to 1912. The plant consisted of at least four steel creosote storage tanks, a wood planing building, an overhead steel conveyor belt system, two creosote weighing vanes located due east of the storage tanks, and a storage building for the treated railroad ties (EJ&E, 1990; Sanborn, 1917; U.S. ACE, 1908). The storage building for the finished product and a 250-foot long, 8-foot high concrete retaining wall (connected to the south edge of the storage building) ran parallel to the EJ&E railroad side tracks.

Available information indicates that the untreated railroad ties were transported by the conveyor to the treating building where they were dipped in vats of creosote. The treated ties were likely transferred to the storage building for future distribution by rail or ship. It is not apparent from the existing data how or where the ties were dried. As a result, the possibility that ties were drip-dried on land used for the creosoting facility operations cannot be eliminated. Based on a review of Sanborn Fire Insurance Maps, the wood treating plant was dismantled sometime after 1917.

3.3.2 Waukegan Manufactured Gas and Coke Plant

In 1927, EJ&E sold the entire property to the William A. Baehr Organization, which in turn sold the property to the North Shore Coke and Chemical Company. Between 1926 and 1928, a coke oven gas plant was designed and constructed under the direction of the William A. Baehr Organization. This gas plant sold their excess gas production to North Shore Gas Company.

The processes and facilities are described below.

The original plant included a large steel and concrete dock for coal unloading located on the western edge of the site along Waukegan Harbor. The western one-third of the site was used for coal storage, from which the coal was transported by drag line and belt conveyors to the coke ovens. The coke ovens consisted of 31 Koppers Company Becker-type ovens, each with a 9.1 ton capacity and an aggregate normal carbonizing capacity of 450 tons per day (Duff & Phelps, 1940).

Before the fall of 1937, some of the gas that was produced was used for the underfiring of the ovens. This practice limited gas production to

3,100,000 cubic feet per day. After the installation of a producer gas plant, which supplied the fuel requirements for the underfiring of the ovens, the daily production was increased to 5,200,000 cubic feet per day. The producer gas that was not used was often blended with the coke oven gas to obtain the desired Btu content and subsequently transmitted to North Shore Gas Company's distribution system (Duff & Phelps, 1940).

Coal tar and ammonia were by-products of the manufactured gas production. The coke company plant included equipment with which gas by-products were extracted and prepared for the market.

In addition to by-product removal, operations at the site included removal of sulfur and naphthalene from the raw gas for gas purification. The gas was treated for sulfur removal on the Coke Company property using equipment owned by North Shore Gas Company (NSG), to whom the Coke Company sold its gas. The purified gas was sent by transmission pipelines for ultimate distributions to the NSG service territory (Duff & Phelps, 1940). The gas purification operations used a liquid sulfur removal process (Thylox) and were conducted at the Thionizer building.

An on-site electric steam generating plant that supplied all of the steam and electricity required for plant operations was owned and operated by the Coke Company (Duff & Phelps, 1940). Steam was generated from two boilers located in the boiler house. The water used to generate the steam was pumped through a 24 inch intake pipe from Waukegan Harbor. In addition, a water well was located at the southwest corner of the boiler house and was completed at a depth of approximately 140 feet below the ground surface (Baehr Organization, 1927).

After the transfer of ownership to GM, the primary function of the plant was to supply coke for a foundry in Saginaw, Michigan. The production of coke oven gas was limited to internal use only due to the conversion of NSG to natural gas in 1947. The gas purification facilities and sulfur removal equipment were dismantled by GM because the coke oven gas was only used on-site.

According to a real estate appraisal description (Real Estate Research Corporation, 1971), the coal preparation and coking portion of the plant consisted of a coal mixing silo, coal preparation building, coke ovens, coke quenching station, domestic screen station, and coke screen and hammer mill building. A 225-foot chimney was located on the southwest corner of the oven. The following structures were located in close proximity or within the by-products building: four cast iron tanks and two gas pumps within the building, a surface tar tank, two steel cooling towers, and cooling coils. To the south of the by-products building was a small tank farm which consisted of three horizontal 15,000-gallon steel tanks, one of which was a tar cooker, as well as two vertical tanks, one for ammonia liquor storage and the other for tar storage.

The plant facilities were dismantled at the direction of OMC in approximately 1972. The specifications for demolition of the coke plant facilities provided for the removal of all of the buildings, smoke stacks, equipment, railroad tracks, and ties (OMC, 1972). According to the specifications, bids were to include the removal of all foundations to 12 inches below grade and the complete removal of the foundations over the coke battery and the two smoke stack bases. The specifications called for removal from the site of any water, oil, tar or residue remaining in the oil or tar storage tanks, and prohibited disposal of oily water, oil, tar, or tar

emulsions in the plant sewer system. Also included in the specifications for the dismantling of the plant was the filling and leveling of all depressed areas such as pits and sumps with incombustible rubble. The site presently is clear of all structures from the coking plant with the exception of the office building in the southeast corner of the site and the above-grade tar tank foundation at the south end of the site. There is evidence that many building foundations are also still present at the site.

3.3.3 OMC Operations

After the demolition and removal of the Coke Plant, OMC used the property for various operations and activities. A data processing building was constructed and is currently maintained on the southeastern portion of the property. Between 1973 and 1989, annual burning permits were obtained from the Illinois EPA for fire prevention and response training for OMC employees. The property has also been used for public parking for special events at the Waukegan public beach. During the winter of 1972-1973, snowmobile performance tests were run on a small track on a portion of the property. OMC's Engineering Department currently performs quality control and durability testing of their products using a tower in the southwest corner of the site (OMC, 1990).

OMC has also used portions of the site for temporary storage of construction materials and semi-trailers. In 1974, the Army Corps of Engineers contracted for the dredging of sand from Lake Michigan. The sands, which were tested and found to contain PCBs (EJ&E, 1990), were placed on the western edge of the site for temporary storage. The dredged spoils are still in-place. Between 1977 and 1980, OMC stored waste oil in two 15,000-gallon above ground storage tanks in the vicinity of the gas producer building.

These tanks have since been removed. Containment areas for these tanks are still evident. During the summer of 1979, four enclosed trailers containing 11,000 gallons of waste oil were parked on the north edge of the site, approximately 50 feet south of Pershing Road (Sea Horse Drive) and 500 feet east of Larsen Marine. Furthermore, two 20,000-gallon tanks which stored gasoline for two to three years in the middle 1970s were located in the center of the site (OMC, 1990). No other information is currently available about these tanks. Larsen Marine has also leased portions of the site for the storage of boats and boat racks.

There is currently an aboveground storage tank farm on the southwestern corner of the site used in OMC's product testing operations. There are nine tanks with a capacity varying from 300 gallons to 20,000 gallons. The stored fuels consist of gasoline, fuel oil, and kerosene (OMC, 1990).

During the latter part of 1990, a contractor to OMC began construction of a new slip to be used for boat servicing. The new slip is located near the northwest corner of the site (Figure 2.3-1). The slip was designed to be 375 feet long by 175 feet wide with a narrowed entrance (Canonie, 1990b). Preliminary plans of the new slip (Canonie, 1991a) indicate that the slip as constructed is approximately 475 feet long. The new slip is intended to replace an existing slip, Slip No. 3, which is located west of the new slip across Waukegan Harbor, and is currently used for the boat servicing operations of Larsen Marine. Slip No. 3 is planned to be filled with PCB-contaminated sediments and subsequently capped as a remedial action for PCB contamination in the Waukegan Harbor.

The new slip constructed at the WCP site includes sheet pile walls and tie-back systems for its north and south borders and a slurry wall at the

eastern end. An existing force main which traverses the new slip site was relocated. Designated contaminated soils (as defined in the Construction Specifications, Canonie, 1990b) excavated during the construction of the new slip were placed at the WCP site in a waste pile intended to meet RCRA guidelines. Soils not defined as designated contaminated soils were placed adjacent to the southeast face of the existing pile of dredge spoils.

3.3.4 Groundwater Quality

The installation of two nests of monitoring wells on the WCP site were part of the New Slip Soil Investigation. The monitoring wells were installed east of the new slip location. Each nest contained a shallow and a deep well with depth intervals of 12.5 to 17.5 feet for the shallow wells and 23 to 28 feet for the deep wells. Samples obtained from the four monitoring wells were analyzed for phenols and PAHs (Canonie, 1990a).

Results of the chemical analyses performed on the water samples indicate that phenolic contamination is detectable in the deep well while PAHs were detected in one of the shallow wells. Detection limits for the PAH analyses of samples from the two deep wells were elevated due to the high concentrations of phenols that were present. The groundwater samples were not analyzed for volatile organic compounds that are commonly associated with coking and coal gasification sites. The range of detected compounds found during the Canonie investigation are summarized in Table 3.3-1.

3.3.5 Surface Water Quality

Extensive studies have been performed to determine the extent of PCB contamination in the Waukegan Harbor. There are no surface waters on the WCP site, but the western border of the site is the Waukegan Harbor. No sampling has been performed to determine if PCBs have migrated on-site.

3.3.6 Soil Quality

As part of the New Slip Soil Investigation (Canonie, 1990a), soil borings were placed in and around the location of the proposed new slip. Laboratory analyses performed on these soil borings indicate high concentrations (up to 27,000 ppm) of total PAHs near the southeast corner of the proposed new slip. PAHs were detected in samples collected to depths of 25 feet below the ground surface. The nature and extent of soil contamination is not fully defined since samples from the soil borings were generally not analyzed for PCBs (i.e., for samples from less than 15 feet in depth) or for volatile organic compounds (VOCs). The range of soil quality found in the Canonie investigation are summarized in Table 3.3-2.

Additional soil investigations were performed in the new slip area by Canonie Environmental in November and December 1990 and January 1991. These investigations were performed to provide additional information for: (1) delineating areas of soils to be placed in the waste pile following excavation of the new slip; and (2) providing information on soil quality in areas affected by the extension of the slip toward the east. As of June 1991 when this document was written, only preliminary information on sampling locations, methods, and results were available (Canonie, 1991a): The final data report for the 1990/1991 soil investigation in the new slip area

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(Canonie, 1991b) was subsequently made available on October 5, 1991. The results of that final report will be incorporated into the Phase I Technical Memorandum of this remedial investigation.

A study performed by the Illinois Environmental Protection Agency (IEPA) in June of 1989 consisted of the collection and analysis of 10 samples from on-site soil borings. Four of the samples were collected near the by-products recovery area, and one of the samples was taken in the gas production area. The remaining five samples were collected at the northern half of the site. The samples were collected between zero and 6 feet in depth. The samples were analyzed for phenols, PAHs, VOCs, pesticides, and metals. Laboratory analyses of the soil samples showed significant concentrations of PAHs, VOCs, and selected metals. Detected PAH concentrations were highest near the tar storage and by-products recovery area. The sample collected near the thionizer building had elevated levels of arsenic and cyanide, and elevated mercury concentrations were reported for the sample collected from the northeast portion of the site. The range of concentrations for detected parameters found in the IEPA investigation are summarized in Table 3.3-3.

3.4 TARGET COMPOUNDS

The nature of past operations at the site was used to define the categories of target compounds (indicator parameters) expected at the WCP site. The compounds of concern or indicator parameters identified at this site are provided in Table 3.4-1.

The compounds were selected based on:

- concentrations detected in previous samples collected at the site;
- suspect contaminants from a manufactured gas/coke plant or creosoting plant operations;
- toxicity of the compounds;
- mobility of the compounds;
- regulatory thresholds for the compounds.

The indicator parameters will be used to evaluate the location and extent of the contamination at the site and to determine action levels for these compounds.

Sampling activities will be conducted in two phases. Activities are summarized in Table 3.4-2. Selected soil and all groundwater samples during Phase I will be analyzed using Contract Laboratory Program (CLP) Routine Analytical Service (RAS) protocols for all TCL organic and TAL inorganic compounds.

During both phases, arsenic and cyanide will be analyzed using CLP SOW Document ILM01.0. Polynuclear aromatic hydrocarbons (PAHs) and acid extractable compounds will all be analyzed using CLP SOW Document OLM01.1. Because the extraction procedure of the current CLP SOW (OLM01.1) is not appropriate for PAH analyses, the old extraction procedure from the CLP SOW 2/88 will be employed to remove potential interferences of phenolic compounds. Groundwater samples from selected wells will be analyzed for PAHs using the low-level PAH method based on an evaluation of the results of initial analyses (using CLP-SOW OLM01.1). The low level PAH analyses will be performed using the SOP "Determination of PAH and Heterocyclics by GC/MS." The list of PAH and acid extractable compounds which will be reported is provided in Table 3.4-3. Target compound list volatile organic compounds

(TCL-VOCs) will be analyzed following procedures specified in CLP-SOW OLM01.1. Additional benzene, toluene, ethyl benzene and xylene analyses will be performed using SW846 method 8020. TCLP analysis of soil will be performed based on the EPA guidelines specified in 55 Federal Register, 126, Friday, June 29, 1990. Soil characteristic testing will be done following appropriate ASTM methods. All general chemistry including corrosivity (pH) analyses of water samples will be done following the appropriate EPA method. Reactivity analyses will be performed by first determining the total cyanide and total sulfide of the sample. If the total concentrations are above action levels (>250 mg HCN/Kg or >500 mg H_2S /Kg), further analyses will be performed using the specific methods for reactive cyanide and reactive sulfide. Section 9 of the QAPP outlines in detail the methods to be used for each analysis. Specific standard operating procedures are provided in Appendix B. Required quantitation limits are listed in Tables 3.4-4 and 3.4-5.

If the Phase I analyses of soil and groundwater samples for a broad range of parameters indicates additional chemical constituents require investigation, such chemicals will be addressed in Phase II. Any such parameters will be identified in the Phase I Technical Memorandum. More detail of Phase I and Phase II activities is provided in Section 6.3 and 6.4 of the Work Plan.

3.5 PROJECT OBJECTIVES

The objectives of the remedial investigation at the WCP site are summarized in Table 3.5-1.

3.5.1 Intended Data Usage

The intended data usage for samples collected at the WCP site is summarized in Table 3.5-2 and Table 3.5-3.

Soil samples will be collected from the test trenches and shallow soil borings for examination using field screening methods. The field screening methods will include field soil classification, visual observations, field oil sheen screening, and field headspace organic vapor screening. These procedures are detailed in the FSP. The field headspace organic vapor screening will be used as an inclusive, qualitative method for assessing soil contamination; i.e., a sample that does not appear to contain chemicals of interest based on other field screening methods but shows headspace organic vapors of greater than 100 parts per million will be included with samples selected for further investigation.

3.5.2 Data Quality Objectives

Data quality objectives (DQO) define and specify the quality of data required for the intended use of the data. The degree of certainty of a data set with respect to precision, accuracy, representativeness, completeness, and comparability is an indication of the data quality.

There are five defined levels of analytical data, outlined below:

- Level I -- Field Screening. The objective of this level of analysis is to generate data to be used in refining sampling plans and determining gross extent of contamination at the site. This

type of data also provides real time monitoring for health and safety.

- Level II -- Field Analysis. The objective of this level of analysis is to provide real-time data for ongoing field activities or when initial data will provide the basis for the selection of additional laboratory analyses. Analyses include the use of an on-site close support laboratory.
- Level III -- Laboratory Analysis. This level of support is designed to provide laboratory analyses using standard EPA-approved procedures other than the current CLP RAS. This level provides data for site characterization, environmental monitoring, confirmation of field data, and to support engineering studies.
- Level IV -- Contract Laboratory Programs (CLP) Routine Analytical Services (RAS). This level provides for the highest level of data quality with full CLP analytical, quality control, and validation procedures in accordance with EPA protocols. The data is used for risk assessment, confirmation of lower level data, and to obtain highly documented data.
- Level V -- Nonstandard Methods. The objective of this level is to provide data not obtained through standard avenues of analytical support. This usually involves modification of existing methods of method development. The level of quality control is usually similar to Level IV data.

A summary table containing the level of DQOs for each group of data is provided in Table 3.5-4.

3.6 SAMPLE NETWORK AND RATIONALE

The sample and analysis program is summarized in Table 3.6-1. Rationale for the selection of samples is provided in Table 3.5-2 and 3.5-3.

3.7 PROJECT SCHEDULE

Table 3.7-1 is a schedule for the RI/FS activities.

SECTION 4
PROJECT ORGANIZATION AND RESPONSIBILITY

4.1 MANAGEMENT RESPONSIBILITIES

The PRP conducting the WCP site RI/FS is North Shore Gas Company. North Shore Gas has overall responsibility for monitoring activities and quality assurance. The PRP Project Manager for North Shore Gas is Patrick Doyle. Mr. Doyle will have the lead role in directing the RI/FS activities.

The U.S. EPA Region V will provide oversight and review for the RI/FS. The EPA regional project manager working on this project is Cindy Nolan. The EPA regional quality assurance officer for this project is Valerie Jones.

The Illinois EPA (IEPA) will also review the RI/FS project. The IEPA project manager working on this project is Scott Moyer.

Barr Engineering Co. (Barr) has been contracted by North Shore Gas to conduct the RI/FS. Barr will be responsible for the study design, field investigation activities, observation of subcontractors (drillers, analytical laboratories, general contractors, and surveyors), data review, and reporting.

Figure 4.1-1 presents an organizational chart of the overall project organization showing the framework for program management.

4.1.1 Barr Engineering Co. Personnel and Responsibility

The Barr Principal-in-Charge of the WCP site RI/FS is Dean Malotky. Mr. Malotky will handle issues of corporate responsibility for the project. The Barr project officer (technical manager), James Langseth, will be responsible for overall management of the project, contracting issues, and assurance that budget, schedule, and work product quality are met.

The Barr project manager (site manager), Michael Relf, will be responsible for preparation of: work plans and technical memoranda; coordination, scheduling, and oversight of project activities with project team members; communication with subcontractors and the PRP project manager; and reporting data and findings generated by the study. The Barr project manager will inform the PRP project manager of all deviations from the QAPP as they occur, including changes in sampling, analyses, quality control, data reduction, and reporting activities.

The project geologist (soil sampling team leader), John Fox, will be responsible for: field inspection of monitoring well and soil boring installation; field screening of soil samples; and classification of soil samples and preparation of well and boring logs.

The project field engineer (test trench survey team leader), William Mielke, will be responsible for field inspection of test trenching investigations and oversight of surveying activities.

The project hydrogeologists, Karlene French and Michael Relf, will be responsible for the performance and analysis of slug tests and pumping tests,

interpretations of groundwater flow patterns, and simulations of groundwater flow and contaminant transport.

The field sampling technicians will be responsible for: well development; groundwater sample collection, preservation, and transportation; sample collection documentation; and sample custody until samples are shipped or turned over to the analytical laboratory.

The project safety officer, Karen Stoller, will be responsible for developing the site safety plan, coordinating the safety training and medical monitoring of investigation personnel, and maintaining safety records.

The Barr quality assurance officer, Mary Mackey, will be responsible for: assisting the project manager in specifying project QA/QC procedures; specifying field sampling and sample analysis methods to be used in the study; communication with the analytical laboratory; auditing the sampling and analytical activities to ensure that the proper techniques and appropriate quality control procedures are followed; data assessment; review of tentatively identified compounds; recommending corrective actions when necessary; and preparing a quality control report.

4.1.2 Laboratory Responsibility and Organization

CH2M Hill Environmental Laboratory (CH2M Hill) will be responsible for the analysis of soil and groundwater samples. Herb Kelly, Organic Division Manager, will be responsible for overall project management, data validation, and quality assurance activities at the laboratory. Additional information on the organizational structure of the laboratory is provided in Figure 4.1-2.

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Barr quality assurance officer, Mary Mackey, will coordinate sampling and analysis activities with CH2M Hill.

Soil characterization procedures will be conducted by Soil Engineering Testing, Inc., Minneapolis, Minnesota.

4.2 QUALITY ASSURANCE ORGANIZATION

Activities of key individuals with respect to quality assurance of sampling, laboratory analyses, quality control, data processing, and data quality review for the project are presented below:

<u>Tasks</u>	<u>Responsible Organization/Personnel</u>
Final review/approval of QAPP	Cindy Nolan, U.S. EPA Region V RPM and Valerie Jones, U.S. EPA Region V QA Officer
QA review and approval reports, SOPs, and field activities; audits of reports, procedures, and activities for identifying, controlling non-conformance for corrective actions	Mary Mackey, Barr Engineering Co., QA Officer
Evidence audits of field records	Mary Mackey, Barr Engineering Co., QA Officer
Data Assessment	Mike Relf, Barr Engineering Co., Project Manager
Performance and System audits of Laboratories Analysis	U.S. EPA Region V CRL
Performance and System audits of field activities	U.S. EPA Region V CRL
Approval of QA Program and laboratory test procedures	U.S. EPA Region V QA Section U.S. EPA Region V CRL

4.5 FIELD OPERATION RESPONSIBILITIES

The responsibilities of key individuals involved in field activities at the site are summarized in the organizational chart in Figure 4.5-1.

SECTION 5
QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA
IN TERMS OF PRECISION, ACCURACY, COMPLETENESS,
REPRESENTATIVENESS AND COMPARABILITY

The overall QA objectives are to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, and reporting that will provide the level of data required for evaluating environmental contaminants. Specific procedures to be used for sampling, chain-of-custody, calibration of field instruments, laboratory analysis, reporting, internal quality control, audits, preventative maintenance, and corrective actions are described in other sections of this QAPP and Field Sampling Plan. This section will address the objectives of precision, accuracy, completeness, representativeness and comparability.

- Precision measures the reproducibility of measurements under a given set of conditions. It is a measure of the variability of a group of measurements compared to an average value.
- Accuracy measures the bias in a measurement system. Possible sources of error are the sampling process, field contamination, preservation, handling, sample matrix, sample preparation, and analysis techniques.
- Completeness is defined as the percentage of measurements made that are judged to be valid measurements.
- Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a

population, parameter variations at a sampling point, or environmental conditions.

- Comparability is a qualitative parameter expressing the confidence with which one data set can be compared to another.

To assess these five parameters, both sampling and analysis options are employed.

5.1 SAMPLING QUALITY ASSURANCE

5.1.1 Field Sampling

Field duplicate (replicate) samples will be collected in the field and submitted to the laboratory to assess the sampling precision. Field duplicate (replicate) samples will be used to assess the combined effects of sample collection, handling, and analysis on data precision. Sampling accuracy for water samples is assessed through evaluation of results of both field and trip blanks. Field blanks will be analyzed to check for procedural factors or ambient conditions at the site that may cause contamination. Trip blanks will be prepared for water VOC samples to check for cross-contamination that may occur during sample storage or shipment. Results of blank samples will be evaluated to assist in the determination of potential false positive sample results.

For water samples, field blank samples and field duplicate (replicate) samples will be collected at the minimum frequency of ten percent or one per day per group of analytical parameters. Trip blank samples for water VOCs will be shipped at the frequency of one (two 40-ml vials) per each shipping

cooler of water VOC samples. For soil samples, field duplicate (replicate) samples will be collected at the minimum frequency of one per 10 or fewer investigative samples. No field blanks will be collected for soils.

The goal of completeness is to ensure that a sufficient amount of valid data are generated. The number of samples obtained and the use of the CLP for laboratory analyses should provide sufficient valid data.

The objective of representativeness is to assess whether the information obtained during the investigation accurately represents the actual site conditions. The sampling network was designed to provide data representative of site conditions. All field sampling activities will be performed following standard sampling techniques and are outlined in the field sampling plan. Factors which will be considered during the evaluation of representativeness include:

- environmental conditions during sampling
- sampling and analytical methodologies
- sampling network - location and number of samples
- analytical parameters.

The use of the standard sampling procedures and recognized field techniques for sampling should make the resulting data comparable to other measurements on similar samples under similar sample conditions.

5.1.2 Field Measurements

Specific procedures used in field measurement of pH, specific conductance, and temperature are presented in SOPs attached to the SAP. Recommended calibration and maintenance procedures are provided in Sections 6 and 8 of the QAPP.

The accuracy of field measurements of pH, temperature, and specific conductance will be addressed through calibration measurements at the beginning of the day and calibration measurement verifications every five samples collected and at the end of the day. The precision of field measurements will be addressed through analysis of field duplicated samples for pH and specific conductance. The frequency of field duplicate analysis will be 10 percent or one per day minimum. If duplicate measurement of pH is not within 0.1 pH units, the pH meter will be recalibrated.

The accuracy of field measurements of VOCs will be addressed through instrument calibration at the time of routine maintenance, and calibration measurement verifications and adjustment at least weekly.

To achieve completeness and sufficient valid data from field measurements, invalid measurements suspected by the samplers to be invalid will be repeated using another instrument or after recalibration of instruments. The goal for completeness for all field activities is 95 percent.

Representativeness and comparability of field measurements is achieved by the use of standard techniques and standard operating procedures to analyze samples.

5.2 LABORATORY QUALITY ASSURANCE

The QA goals for RAS are established under CLP guidelines, as stated in the EPA CLP RAS Statement of Work (SOW) for Organics Analysis (OLM01.0) and the EPA CLP RAS Statement of Work for Inorganics Analysis (ILM01.0). Precision and accuracy requirements for RAS analyses are specified in the CLP SOWs. Additional parameters will be analyzed following the laboratory procedures for non-CLP analyses (Appendix B). Precision and accuracy requirements for non-CLP analysis are specified in the analytical procedure. Data which meet the precision and accuracy requirements of the CLP SOWs and the analytical procedure will be sufficient to meet the objectives of this project. Data that do not meet precision and accuracy requirements will be assessed on a case by case basis to determine if they are usable to support project objectives.

Data completeness can be quantified during data assessment. The goal of the monitoring program is to achieve 100 percent data completeness. The goal for laboratory data completeness on this project is 95 percent. All data that meet QA/QC acceptance criteria will be used in the decision making process, even if the data set as a whole or for any analytical fraction does not meet the completeness goal.

Representativeness assesses the degree to which the data represent actual site conditions. The use of standard sampling techniques and the design of the sampling network provide data representative of site conditions. The use of standard procedures to analyze representative samples provides comparable data.

SECTION 6

SAMPLING PROCEDURES

6.1 INTRODUCTION

The general objective of sampling is collection of a sample representative of conditions at the monitoring point. The project-specific sampling objectives will be established by the project manager before field activities begin. Project specific objectives and facility conditions will dictate the type of sample collected (soil, sediment, groundwater, waste or surface water), sampling procedures, and sampling equipment. Specific field sampling procedures are provided in the Field Sampling Plan (FSP).

Prior to visiting the site, all team members will review the RI/FS Work Plan, the FSP, the Site Health and Safety Plan, and the QAPP. If necessary, project team meeting will also be conducted with the purpose of clarifying the tasks and objectives of the project, and reviewing available site information.

Before sampling commences, a visual evaluation of the site will be conducted. The visual evaluation will include observation of sampling points, routes of access, key landmarks, and assessment of potential hazards. During sample collection, the FSP and QAPP will be followed in detail, and the sampling procedures and pertinent observations will be documented. Any changes to the existing field sampling plan or QAPP will be discussed with the U.S. EPA project manager.

6.2 EQUIPMENT

All drilling and groundwater sampling, developing and purging equipment will be carefully cleaned before use. All drilling equipment will be steam cleaned prior to use and between soil borings. All groundwater sampling equipment except bailers will be cleaned with soap and water and rinsed with tap water prior to use. Bailers will be cleaned in the laboratory with soap and water and rinsed sequentially with tap water and distilled water. Bailers will then be baked at 200°C for at least one hour. The bailers will be transported to the field wrapped in aluminum foil. Each specially prepared bailer will be used to collect samples from only one well before being returned to the laboratory for cleaning.

6.3 SOIL SAMPLING

Section 3 of the FSP discusses the detailed procedures, criteria and guidelines for sampling and sampling point selection. These are to be followed for the collection of soil samples for evaluation of physical and chemical soil characteristics.

6.4 GROUNDWATER SAMPLING

Section 3 of the FSP discusses the detailed procedures, criteria, and guidelines for sampling. These are to be followed for the collection of samples for evaluation of chemical and physical characteristics.

Organic-free water for use in trip blanks and field blanks will be provided by the laboratory. Daily blanks are analyzed by the lab to verify the water is free of any contaminants.

Trip blanks will be prepared by the laboratory using organic free deionized water. One trip blank for VOC's will travel with each cooler containing VOC sample containers and/or samples. The trip blank will remain in the cooler unopened until received at the lab for analysis.

Field blanks (rinsate blanks) will be organic-free water provided by the laboratory, and will be prepared by collecting the organic free water in the sample container after it has been poured over previously decontaminated equipment (i.e., laboratory cleaned bailer). Field blanks will be analyzed following the same procedures as the samples.

Field duplicate samples will be collected by alternating the duplicate containers with the sample containers. Duplicate samples will be analyzed in the field for pH and specific conductance and will be submitted blindly to the laboratory designated with the letter "M" prefix followed sequentially by the number (M-1, M-2, . . .).

6.5 SAMPLE IDENTIFICATION

The Field Sampling Plan addresses the assigning of unique sample identifiers for each sample generated at the site.

6.6 SAMPLE CONTAINERS, PRESERVATION AND HOLDING TIMES

These are outlined in Table 6.6-1. All sampling containers used for laboratory analysis will be supplied by the subcontracting laboratory. Only new sample bottles are used and are purchased from a commercial supplier. All sample containers for organic parameters and cations are quality control checked by the container supplier prior to distribution. QC documentation is

acquired from the supplier and stored by the laboratory. Examples of QC documents are provided in Appendix C. In addition, to ensure that sample containers are free from contamination, a bottle blank will be analyzed prior to sampling for each lot of sample containers provided by the laboratory for organic parameters and metals. Bottle blanks will be identified to correspond with samples collected during each phase of the project (i.e., BB/PI-1 = Bottle Blank Phase I Lot #1). For inorganic wet chemistry and some soil cations, the laboratory performs an in-house quality control check on 1 percent of all these bottles. For additional detail, the standard operating procedure for preparing sample kits is provided in Appendix B.

6.7 FIELD ANALYSES AND FIELD SCREENING TECHNIQUES

Specific conductance, temperature, and pH will be measured in the field immediately prior to collection of water samples. These measurements will be collected as part of the stabilization test. The following instruments or their equivalent will be used for analyses in the field:

- Orion Research Model 407A pH meter
- YSI Model 33 specific conductance meter (includes temperature measurement)

The specific standard operating procedures for the calibration of the pH and conductivity meter and sample analysis are provided in Appendix A.

The field screening techniques for soils containing coal tar are as follows: (1) Visual Examination; (2) Oil Sheen; (3) Odor; and (4) Headspace Organic Vapor Screening. The results of these four screening procedures will

be used to determine the gross level of PAH contamination of the soil sample. The standard operating procedure for this is provided in Appendix A.

6.8 SAMPLE TRANSPORTATION

Samples will be shipped to the laboratory via a next-day delivery service. All samples will be shipped to the laboratory by overnight carrier within 24 hours of sampling. Shipping receipts will be retained for all samples.

SECTION 7

SAMPLE CUSTODY

7.1 INTRODUCTION

It is U.S. EPA and Region V Policy to follow the U.S. EPA Region V sample custody, or chain of custody protocols as described in "NEIC Policies and Procedures", EPA-330/9-78-DD1-R, Revised June 1985. This custody is in three parts: Sample collection, Laboratory analysis, and Final evidence files. Final evidence files, including all originals of laboratory reports and purge files, are maintained under document control in a secure area.

A sample or evidence file is under your custody if they:

- are in your possession;
- are in your view, after being in your possession;
- are in your possession and you place them in a secured location; or
- are in a designated secure area.

7.2 CHAIN-OF-CUSTODY IN THE FIELD

The field sampler will be responsible for custody of samples until they are properly dispatched to the laboratory or turned over to an assigned custodian. The field sampler will ensure that possession or sight of sample containers is maintained at all times or that the containers are stored in a securely locked area. A chain-of-custody record will identify the samples in the transfer container and to summarize the analyses to be carried out on each sample. A chain-of-custody form is shown in Figure 7.2-1.-

The chain-of-custody procedures will apply to all samples collected. All field entries will be completed in indelible ink. All errors will be corrected by drawing a single line through the incorrect information. All corrections will be signed and dated by the individual making the correction. The original chain-of-custody record and one copy for retention by the laboratory will be sealed in a waterproof container and shipped inside the sealed transportation case. A second copy of the record will be retained by the sampling team, and the third copy will be retained by the Barr Engineering Co. quality assurance officer.

Upon collection of the samples, all sample containers will be tightly capped and will be labeled with project identification and sample identification/location, and immediately placed into a cooler containing ice. An example of the sample label is provided in Figure 7.2-2.

All sample coolers will be sealed with tamper-proof security tape signed and dated by the individual sealing the cooler. An example of the custody seal is provided in Figure 7.2-3.

The addresses of the consignee and consignor will be printed on the outside of the transfer container or attached firmly thereon by cards and labels. As necessary, warning and descriptive labels will be attached to the transfer container.

7.2.1 Field Logs

A bound field log will be maintained throughout the monitoring program. The following information will be recorded in the field log:

- Date
- Weather
- Personnel present, including visitors and observers and the purpose of their visit
- locations sampled
- Equipment used
- Level of protection
- Non-conforming events during sampling
- Significant communications
- Observations made
- Field equipment calibration record
- Photographs taken
- Log-keepers signature at the end of the day's notes

7.3 CHAIN-OF-CUSTODY SAMPLES IN THE LABORATORY

Samples are physical evidence and are handled according to certain procedural safeguards. In some types of legal proceedings, a showing to the court that the laboratory is a secure area may be all that is required for the analyzed evidence to be admitted. However, in some cases, a court may require a showing of the hand-to-hand custody of the samples while they were at the laboratory. In the event that the court requires such a comprehensive chain-of-custody demonstration, the laboratory must be prepared to produce documentation that traces the in-house custody of the samples from the time of receipt to completion of the analysis. For this work, documentation of in-house chain-of-custody is a routine requirement.

Once samples are received in the laboratory, they are placed in a secure storage area to which only laboratory personnel have access. The general

criterion used to determine if a sample is in custody at CH₂M Hill's Laboratory is if:

- It is actually in an analyst's possession; or
- It is in the analyst's view after being in his or her physical possession; or
- It is in a secure area.

To satisfy these custody provisions, the laboratory implements the following procedures:

- Samples are stored in a secure area.
- Access to the laboratory is through a monitored reception area. Other access doors to the laboratory are kept locked.
- Visitors must sign in at the reception area and are escorted while in the laboratory.
- Samples remain in the secure storage area until they are removed for sample preparation or analysis.
- Refrigerators, freezers, and other sample storage areas are locked during nonwork hours.
- Only the sample coordinators and supervisors have keys to the sample storage area(s).

- After a sample has been removed from storage by the analyst, the analyst is responsible for the custody of the sample. Each analyst must return the samples to the storage area before the end of the working day.
- For clients requiring internal chain-of-custody, a sample control record is completed and placed in the case file. Access is through the sample custodian, who maintains this record until the analyses are completed and the data is released.

7.4 CUSTODY OF EVIDENCE FILE

Until completion of the project, all correspondence, laboratory reports, and data will be maintained in Barr Engineering project files. All original laboratory reports and field data are maintained in their original format and stored separately from working copies of these reports. The Barr project manager and Barr Information Services Specialists will maintain the project file. Following completion of the project, the evidence file will be relinquished to the U.S. EPA for archiving.

The evidence file will contain a document inventory listing all contents of the file. All sampling and analytical correspondence will be included. Other items to be included are: field notes, field log data sheets, airbills, chain-of-custody records, analytical deliverables, QA reports.

SECTION 8
CALIBRATION PROCEDURES AND FREQUENCY

8.1 CLP/ROUTINE ANALYTICAL SERVICES (RAS)

Calibration procedures and frequency for CLP RAS are found in the EPA Contract Laboratory Program Statements of Work for Organics (OLM01.0) and Inorganics (ILM01.0).

8.2 NON-CLP ANALYTICAL SERVICES

Every instrument used to analyze samples must pass the calibration criteria established in the appropriate SOP. Initial calibration criteria for instrument linearity, sensitivity, resolution, and deactivation must be met before samples can be analyzed. Sustained performance is monitored periodically during analyses by the use of continuing calibration check standards.

Instrument calibration procedures are described in the following sections of the analytical SOP:

1. Volatiles 8010/8020: Section 6.0 Initial Calibration
Section 7.0 Continuing Calibration
2. Low Level PAHs: Section 9.0 Tuning and GC/MS Mass Calibration
Section 10.0 Instrument Calibration
Section 11.0 Continuing Calibration
3. BOD: Section 6.0 Calibration of DO Meter

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8.3 FIELD INSTRUMENTS

Calibration procedures and frequency for field instrumentation are described in the standard operating procedures (SOP's) for those instruments and analyses. The SOP's are provided in Appendix A.

SECTION 9

ANALYTICAL PROCEDURES

9.1 ROUTINE ANALYTICAL SERVICES

Analytical procedures for CLP RAS analysis are found in the EPA Contract Laboratory Program Routine Analytical Services Statements of Work for Organics (OLM01.0) and Inorganics (ILM01.0). These procedures will be used for PAH, acid extractable compounds, TCL-VOCs, metals, and cyanide and whenever the work plan requires Full-CLP-RAS to be performed.

9.2 NON-CLP ANALYTICAL SERVICES

Non-CLP analytical services include: analysis of aromatic volatile organics (BTEX), total suspended solids, oil and grease, biochemical and chemical oxygen demand, TOC, flashpoint, corrosivity (pH), reactivity, and low level PAHs, if required. TCLP analyses will be performed based on the EPA guidelines specified in 55 Federal Register 126, Friday June 29, 1990. The standard operating procedure for the non-CLP analyses are provided in Appendix B.

Soil characterization procedures will be conducted by Soil Engineering Testing, Inc., Minneapolis, Minnesota. The procedures used for characterization of soil are specified in Table 9.3-1.

9.3 FIELD ANALYSIS

Analytical procedures for field measurements of pH, specific conductivity, and temperature are provided in Appendix A. The procedure for field screening techniques for soils containing coal tar is also provided in Appendix A.

SECTION 10
INTERNAL QUALITY CONTROL CHECKS

10.1 ROUTINE ANALYTICAL SERVICES

Internal quality control procedures for CLP RAS analysis are found in the EPA Contract Laboratory Program Statements of Work for Organics (OLM01.0) and Inorganics (ILM01.0).

10.2 NON-CLP ANALYTICAL SERVICES

For non-CLP analysis, internal quality control checks are described in the specific analytical procedure.

Internal QC checks are described in the following sections of the SOP:

1. Volatiles 8010/8020: Section 8.0
2. Low Level PAHs: Section 12.0
3. BOD: Section 5.0
4. COD: Section 9.0
5. TSS: Section 5.0
6. Oil and Grease: Section 5.0

10.3 FIELD ANALYSIS

Field analyses will be performed on-site and will not involve samples that are collected and retained. The primary QA/QC objective is to obtain reproducible measurements to a degree of accuracy consistent with limits imposed by analytical methodologies used and with the intended use of the

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data. Quality control procedures will be limited to checking the reproducibility of measurements by taking multiple readings (10 percent duplicate) and by calibration of instruments (where appropriate).

The accuracy of field measurements of pH, temperature, and specific conductance will be addressed through calibration measurements at the beginning of the day and calibration verification for every five samples collected and at the end of the day.

SECTION 11

DATA REDUCTION, VALIDATION, AND REPORTING

11.1 DATA REDUCTION

11.1.1 Laboratory Analysis

Data reduction procedures for CLP RAS analysis are specified in the U.S. EPA CLP RAS Statements of Work for the CLP RAS organic target compound list compounds (OLM01.0) and the CLP RAS inorganic target analyte list compounds (ILM01.0).

Analysts are responsible for the reduction of non-CLP raw data when such steps are required to produce the correct data format for reporting. Data reduction may be done manually or through one of a number of computer programs used in the laboratory.

When non-CLP data has been acquired for a sample, the initial review is done by the analyst. This review covers sample identification, check of analyses requested against the LIMS record, review of procedures and notebook data, checking of calculations done, QC data, and checking for transcription errors. Following this review, the sample data with supporting information as required may be reviewed by a peer, but is always reviewed by the analyst's supervisor.

The supervisory review includes checking for correct analyses performed, correct identification, proper choice of method, correct calculations, investigation of all related quality control data, and correct transcription of all data.

The ultimate responsibility for the analytical results lies with the division manager who makes the final review. All out-of-control conditions noted are reviewed by the laboratory QA coordinator, (LQAC) and the division manager. Decisions concerning these out-of-control conditions are made jointly by the division manager and the LQAC.

After data has been entered into the reporting system, a draft report is reviewed by the supervisor or division manager. This review includes a check of holding times met, correct analysis and report dates, and correct reporting units, as well as a review of results, quality control data, and transcription. At any point in the review process, if an error is found, the analyst has the responsibility for investigating the problem and initiating the correction. During the review process, points which should be brought to the client's attention, such as missed holding times or matrix effects noted in samples, are noted for inclusion in the case narrative or cover letter.

The LQAC routinely checks approximately 90 percent of completed data packages before submittal to the client.

11.1.2 Field Measurements

Raw data from field measurements and sample collection will be recorded on the field data sheets. All specific conductivity data will be corrected to 25°C. The data will be transferred from field data sheets to a computer database and output in a spreadsheet format.

11.2 DATA VALIDATION

11.2.1 Laboratory Analysis

Data validation will be done initially by the subcontracting laboratory prior to reporting. Data validation is part of the review process whereby data are inspected and either accepted or rejected based on a set of criteria. Before analytical results are reported to the client, this review and approval process must be completed.

The analyst has the initial responsibility for proper instrument conditions and calibration, for the data meeting all acceptance criteria, and for all calculations being accurate. After proper instrument conditions and calibration are verified, data generated is validated on the basis of accuracy, precision, and how the data compare with the established limits of detection. Attention is paid to possible outliers. Statistical tests are used to ensure that if data are rejected, it is done with a high level of confidence.

Laboratory analysis reports will generally be submitted to Barr Engineering Co. within 4 weeks after receipt of samples.

Data will be evaluated by the Barr Engineering QA officer to determine if it meets project requirements. Data validation procedures will be consistent with the U.S. EPA documents for Laboratory Data Validation - Functional Guidelines for Evaluating Organic Analyses and Inorganic Analyses. The specific requirements to be checked during data validation are listed below:

1. Holding Times
2. GC/MS Tuning
3. Method Blanks
4. Calibration
5. Surrogate Recovery
6. Matrix Spike/Matrix Spike Duplicate
7. Field Duplicates
8. Field Blanks
9. Overall Data Assessment

Upon completing the validation procedure for all data, a quality control review report will be compiled and submitted to the client and regulatory agencies overseeing the project.

11.2.2 Field Measurements

Data validation of field measurements will be the responsibility of the Barr QA Officer. The application of statistical evaluation procedures are not appropriate for field measurements. Field notes will be checked to verify that specific QC procedures including instrument calibration were performed. Data will be proofed from field notebooks against computer spreadsheets. Calculations performed by the sampler will be rechecked.

11.3 DATA REPORTING

11.3.1 Laboratory Analysis

CH₂M Hill Laboratories recognize the need for providing different levels of QC documentation to meet the needs of clients with different requirements

for project specific QA/QC, report content, format, and documentation of data quality. To meet these needs, the laboratories offer three standard levels of reporting and QC documentation as follows:

Level 1 - Level 1 involves reporting of the analytical results and limited QC data summaries including surrogate recoveries for organic analyses and method blank results for all analytical fractions.

Level 2 - Level 2 involves the reporting of all QC and calibration data summaries for a client or project specific batch of samples. All QC samples are scheduled and analyzed relative to a project or client sample delivery group rather than the total number of samples analyzed by the laboratory. This level of reporting documentation insures that acquired summary data is pertinent to the samples from a given client or project.

Level 3 - Level 3 involves the reporting of all QC and calibration data summaries along with the hardcopy documentation or all raw data for both samples and QC samples. All QC samples are scheduled and analyzed relative to the client or project sample delivery group. All instrument output and related documentation will be provided in hardcopy form. This level provides the standard data package for all routine analytical services defined under the Contract Laboratory Program.

Data generated from samples for this site will be reported by the laboratory using a Level 3 reporting format.

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Data will be entered into a computer database and output in spreadsheet format to be used in reports. An example spreadsheet is presented in Table 11.3-1.

11.3.2 Field Measurements

Data will be summarized and output on computer spreadsheets along with the laboratory data for reports.

SECTION 12
PERFORMANCE AND SYSTEM AUDITS

12.1 EXTERNAL AUDITS

12.1.1 CLP Laboratories

CLP Laboratories analyze quarterly performance evaluation samples and are inspected through on-site technical/evidence audits. U.S. EPA Region V, Central Regional Laboratory (CRL) is responsible for these performance and system audits. The necessity and the frequency of non-CLP external audit will be determined by the LSSS, CRL, Region V.

12.1.2 Field Audits

Region V CRL will be responsible for external field audits and reserve the right to audit during the course of the sampling event.

12.2 INTERNAL AUDITS

12.2.1 Laboratory Audits

Internal audits of the laboratory are conducted in two phases.

The first phase is conducted by the District Quality Assurance Manager at least once a year. This is usually a 2-day systems audit which covers all sections of the laboratory. An audit report is issued within 2 weeks of completion. The LQAC has the responsibility for coordinating all responses

to the audit finding and for following up on the required corrective action. A follow-up audit is made when deemed necessary by the District QA Manager.

The second phase consists of quarterly audits performed by the LQAC. These are day-long audits, and are concentrated on specific areas that are deemed problem areas by the LQAC. An audit report is issued at the completion of the audit. Responses and follow-up corrective action to the audit findings are required, and are monitored by the LQAC.

All audit reports are issued to management and circulated to all staff. Copies are filed with the District Quality Assurance Manager and the LQAC.

12.2.2 Field Audits

Field performance audits are conducted semi-annually by the Barr QA Officer to evaluate the execution of sample identification and control, chain-of-custody procedures, field documentation, training, and sampling operation. Audits evaluate compliance with the procedures outlined in the QAPP and FSP. A field audit checklist is provided in Appendix A.

12.2.3 Other Internal Audits

Barr through its QA Officer will be responsible for conducting internal performance and system audits of its subcontracting laboratories. Audits will be completed at a frequency of once per year.

Internal audits of the laboratory will assess the compliance with the laboratory portions of the QAPP; for analyses run under Contract Laboratory Program (CLP) protocol, the RCRA Laboratory Audit Inspection Form from the

"RCRA Laboratory Audit Inspection Guidance Document, September 1988" and the current revisions of the Organic and Inorganic Statements of Work for the CLP will guide the internal audit.

The on-site inspection will cover:

- A. Qualifications of the laboratory personnel and the organizational structure of the laboratory
- B. Procedures for maintaining laboratory supplies and equipment
- C. Procedures for equipment calibration
- D. Procedures for sample handling
- E. Quality Control procedures
- G. Procedures for data handling, reporting, record keeping

The on-site visit will also serve as a mechanism for discussing weaknesses identified through review of data deliverables. Lastly, the on-site visit will allow Barr to determine if the laboratory has implemented the recommended and/or required corrective actions, with respect to quality assurance, made during any previous on-site visits.

SECTION 13
PREVENTIVE MAINTENANCE

13.1 LABORATORY INSTRUMENTS

CH₂M Hill Laboratory is equipped with advanced instrumentation for fast, accurate, and precise analysis of water, soil/sediment, and air samples.

Maintenance Schedule

Preventative maintenance, such as lubrication, source cleaning, and detector cleaning, is performed according to the procedures delineated in the manufacturer's instrument manuals.

The frequency of preventative maintenance varies with different instruments. Routine maintenance performed includes cleaning and/or replacement of various instrument components. In general, the frequency recommended by the manufacturer is followed. In addition to the regular schedule, maintenance is performed as needed. Precision and accuracy data are examined for trends and excursions beyond control limits to determine evidence of instrument malfunction. Maintenance is performed when an instrument begins to degrade as evidenced by the degradation of peak resolution, shift in calibration curves, decreased ion sensitivity, or failure to meet one or another of the quality control criteria.

Instrument maintenance logbooks are maintained in the laboratory at all times. The logbook contains a complete history of past maintenance, both routine and nonroutine. The nature of work performed, the date, and the signature of the person who performed the work are recorded in the logbook.

Preventative maintenance is scheduled according to each manufacturer's recommendation. Instrument downtime is minimized by keeping adequate supplies of all expendable items on hand. Expendable items are those with an expected lifetime of less than one year.

Routine instrumentation preventive maintenance is handled by the instrument operator. Repair maintenance is performed by a full-time electronics technician, or by the manufacturer's service personnel.

13.2 FIELD INSTRUMENTS

Well stabilization field instruments are calibrated twice daily. Initial calibration will be conducted before sampling occurs. If results indicate meter malfunctioning, corrective actions will be taken (i.e., clean probe, etc.). A final calibration will be conducted at end of the monitoring day. If results indicate instrument not properly calibrated sample results from that day will be flagged with a qualifier.

To limit the potential for equipment to fail in the field, additional batteries and/or, (if economically feasible) replacement equipment is taken to the site.

SECTION 14
SPECIFIC ROUTINE PROCEDURES TO ASSESS DATA
PRECISION, ACCURACY AND COMPLETENESS

Precision, accuracy, and completeness are defined in Section 5 of the QAPP. Equations for calculating precision, accuracy, and completeness are detailed below.

Precision is a measure of the reproducibility of field sampling and laboratory analyses. Both field duplicates (replicates) and laboratory duplicates are analyzed to determine data precision. The results are reported as the relative percent difference (RPD) and are calculated by:

$$RPD = \frac{D1 - D2}{(D1 + D2)/2} \times 100\%$$

where:

D1 = concentration of first duplicate

D2 = concentration of second duplicate

The accuracy of analytical results is a measure of the agreement between an experimental determination of the true value of the parameter being measured. Spike sample analyses are used to determine the accuracy of analyses. A known quantity of the constituent of interest is added to a sample and analyzed. The amount of spiked compound recovered by analysis is compared to the amount added. Percent recovery (%R) is calculated by:

$$\%R = \frac{SSR - SR}{SA} \times 100\%$$

SSR = quantity measured in spike sample
SR = quantity measured in unspiked sample
SA = quantity of spike added

The completeness of data from a sampling program is interpreted as the percentage of valid data obtained compared to the amount that was expected to be obtained.

$$\text{Completeness} = \frac{\text{Data values useable}}{\text{Total data values obtained}} \times 100\%$$

14.1 QUALITY CONTROL (QC) REVIEW

Data will be assessed by Barr Engineering Co. for the following QC elements:

- Sample Holding Times
- Accuracy of Spiked Samples
- Precision of Duplicate Samples
- Instrument Calibration
- Blank Results
- Surrogate Recovery
- Comparison with Historical Data

■ Potential False Positive Results

A Quality Control Review Form will be completed for each laboratory report to summarize this data evaluation. An example of the Quality Control Review Form is provided in Figure 14.1-1.

All quality control reviews will be discussed with the project manager and the MPCA to determine the useability of the data.

SECTION 15
CORRECTIVE ACTION

When problems are encountered during either field or laboratory activities which could effect data quality or when the QC data exceed the acceptance criteria, corrective actions will be implemented. Once a problem has been identified, the project manager will be notified. U.S. EPA staff will also be informed. Informal or formal discussions will take place to determine the effect on the data and possible corrective actions. Possible corrective actions might include:

1. Reanalysis of samples
2. Recollection and analysis of samples

It is the responsibility of the Barr QA Officer to initiate and implement the corrective action process once a problem is identified in the field or at the laboratory. The problem will be documented on a Corrective Action Form provided in Figure 15.1-1. Copies of the form will be sent to the Barr project manager and EPA project manager. All action will be documented and included in the evidence file.

SECTION 16
QUALITY ASSURANCE REPORT TO MANAGEMENT

While a project is in progress, a quality assurance summary report will be provided to the project manager during each phase of the project. Monthly progress reports for the project will be submitted to the EPA. Any QA concerns will be included in this monthly report. The overall quality assurance performance for this project will be addressed in the final report. Any non-compliance with the QAPP or project concerns will be immediately reported to the U.S. EPA.

The contents of the QA section of the report will include the following elements:

- Performance and systems audits conducted during the project
- Data validation narrative summary and data quality assessment
- QA problems and corrective action
- Proposed changes to the QAPP and/or Field Sampling Plan
- Qualified data summarized in tables

Tables

TABLE 3.3-1

Summary Table
Existing Groundwater Quality Data (1)

Compound	Range of Reported Concentrations (ug/L)	Detection Limit (2) (ug/L)	Method (3)

SEMIVOLATILES			
PAH Compounds			
Acenaphthene	BDL-85	10	
Acenaphthylene	BDL	10	
Anthracene	BDL	10	
Dibenzofuran	BDL-47J	10	
Fluoranthene	BDL	10	
Fluorene	BDL-43J	10	
Naphthalene	BDL-1100	10	
2-Methylnaphthalene	BDL-52	10	
Phenanthrene	BDL-38J	10	
Pyrene	BDL	10	
Benzo(a)Anthracene (c)	BDL	10	
Benzo(a)Pyrene (c)	BDL	10	
Benzo(b)Fluoranthene (c)	BDL	10	
Benzo(g,h,i)Perylene (c)	BDL	10	
Benzo(k)Fluoranthene (c)	BDL	10	
Chrysene (c)	BDL	10	
Dibenzo(a,h)Anthracene (c)	BDL	10	
Ideno(1,2,3,c,d)Pyrene (c)	BDL	10	
Phenolic Compounds			
2-Methylphenol (O-Cresol)	BDL-27000	10	
4-Methylphenol (P-Cresol)	BDL-100000	10	
2-Chlorophenol	BDL	10	
2,4-Dichlorophenol	BDL	10	
2,4-Dimethylphenol	BDL-9000	10	
2,4-Dinitrophenol	BDL	50	
4,6-Dinitro-2-Methylphenol	BDL	50	
2-Nitrophenol	BDL	10	
4-Nitrophenol	BDL	50	
Pentachlorophenol	BDL	50	
Phenol	BDL-160000	10	
2,4,5-Trichlorophenol	BDL	50	
2,4,6-Trichlorophenol	BDL	10	
Pesticides			
Aldrin	NT		
BHC, Alpha-	NT		
BHC, Beta-	NT		
BHC, Delta-	NT		
BHC, Gamma- (Lindane)	NT		

TABLE 3.3-1

Summary Table
Existing Groundwater Quality Data (1)

Compound	Range of Reported Concentrations (ug/L)	Detection Limit (2) (ug/L)	Method (3)
4,4'-DDD (P,P'-)	NT		
4,4'-DDE (P,P'-)	NT		
4,4'-DDT (P,P'-)	NT		
Dieldrin	NT		
Endosulfan I	NT		
Endosulfan II	NT		
Endosulfan Sulfate	NT		
Endrin	NT		
Endrin Aldehyde	NT		
Heptachlor	NT		
Heptachlor Expoxide	NT		
Toxaphene	NT		
4-Chloroaniline	BDL	10	
2-Nitroaniline	BDL	50	
3-Nitroaniline	BDL	50	
4-Nitroaniline	BDL	50	
Chlordane, Alpha	NT		
Chlordane, Gamma	NT		
Endrin Ketone	NT		
Methoxychlor	NT		
PCBs			
Arochlor-1016	NT		
Arochlor-1221	NT		
Arochlor-1232	NT		
Arochlor-1242	NT		
Arochlor-1248	NT		
Arochlor-1254	NT		
Arochlor-1260	NT		
Other Semivolatiles			
Benzoic Acid	BDL	50	
Benzyl Alcohol	BDL	10	
Benzyl Butyl Phthalate	BDL	10	
Bis(2-Chloroethoxy)Methane	BDL	10	
Bis(2-Chloroethyl)Ether	BDL	10	
Bis(2-Chloroisopropyl)Ether	BDL	10	
Bis(2-Ethylhexyl)Phthalate	BDL	10	
Benzidine	NT		
4-Bromophenyl Phenyl Ether	BDL	10	
2-Chloronaphthalene	BDL	10	
4-Chlorophenyl Phenyl ether	BDL	10	
1,2-Dichlorobenzene	BDL	10	

TABLE 3.3-1

Summary Table
Existing Groundwater Quality Data (1)

Compound	Range of Reported Concentrations (ug/L)	Detection Limit (2) (ug/L)	Method (3)
1,3-Dichlorobenzene	BDL	10	
1,4-Dichlorobenzene	BDL	10	
3,3'-Dichlorobenzidine	BDL	20	
Diethylphthalate	BDL	10	
Dimethylphthalate	BDL	10	
2,4-Dinitrotoluene	BDL	10	
2,6-Dinitrotoluene	BDL	10	
Di-N-Butylphthalate	BDL	10	
Di-N-Octylphthalate	BDL	10	
1,2-Diphenylhydrazine	NT		
Hexachlorobenzene	BDL	10	
Hexachlorobutadiene	BDL	10	
Hexachlorocyclopentadiene	BDL	10	
Hexachloroethane	BDL	10	
Isophorone	BDL	10	
Nitrobenzene	BDL	10	
N-Nitrosodiphenylamine	BDL	10	
N-Nitroso-Di-N-Propylamine	BDL	10	
N-Nitrosodimethylamine	NT		
1,2,4-Trichlorobenzene	BDL	10	
P-Chloro-M-Cresol	BDL	10	

BDL- Below detection limit

Blank- Blank contamination in samples

NT- Not tested

J- Below quantitation limit

(1) Canonie Environmental Inc., 1990a.

(2) Minimum detection limits presented (where available)

(3) Information on analytical methods generally not provided in referenced data report.

TABLE 3.3-2

Summary Table
Existing Soil Quality Data, New Slip Investigation (1)

Compound	Range of Reported Concentrations (ug/kg)	Detection Limit (2) (ug/kg)	Method (3)

SEMIVOLATILES			
PAH Compounds			
Acenaphthene	BDL-1200000	410	
Acenaphthylene	BDL-440000	410	
Anthracene	BDL-880000	410	
Dibenzofuran	BDL-960000	xx	
Fluoranthene	BDL-2900000	410	
Fluorene	BDL-1700000	410	
Naphthalene	BDL-12000000	790	
2-Methylnaphthalene	BDL-2300000	xx	
Phenanthrene	BDL-6400000	410	
Pyrene	BDL-2200000	410	
Benzo(a)Anthracene (c)	BDL-770000	410	
Benzo(a)Pyrene (c)	BDL-760000	410	
Benzo(b)Fluoranthene (c)	BDL-770000	410	
Benzo(g,h,i)Perylene (c)	BDL-120000	410	
Benzo(k)Fluoranthene (c)	BDL-770000	410	
Chrysene (c)	BDL-800000	410	
Dibenzo(a,h)Anthracene (c)	BDL-5700	410	
Ideno(1,2,3,c,d)Pyrene (c)	BDL-130000	410	
Phenolic Compounds			
2-Methylphenol (O-Cresol)	BDL-34000	xx	
4-Methylphenol (P-Cresol)	BDL-79000	xx	
2-Chlorophenol	BDL	410	
4-Chloro-3-Methylphenol	BDL	410	
2,4-Dichlorophenol	BDL	410	
2,4-Dimethylphenol	BDL-8700	790	
2,4-Dinitrophenol	BDL	2000	
4,6-Dinitro-2-Methylphenol	BDL	2000	
2-Nitrophenol	BDL	410	
4-Nitrophenol	BDL	2000	
Pentachlorophenol	BDL	2000	
Phenol	BDL-220000	790	
2,4,6-Trichlorophenol	BDL	410	
Pesticides			
Aldrin	BDL	19	
BHC, Alpha-	BDL	19	
BHC, Beta-	BDL-8800	19	
BHC, Delta-	BDL	19	

TABLE 3.3-2

Summary Table
Existing Soil Quality Data, New Slip Investigation (1)

Compound	Range of Reported Concentrations (ug/kg)	Detection Limit (2) (ug/kg)	Method (3)

BHC, Gamma- (Lindane)	BDL-49000	16	
4,4'-DDD (P,P'-)	BDL	38	
4,4'-DDE (P,P'-)	BDL	38	
4,4'-DDT (P,P'-)	BDL	38	
Dieldrin	BDL	38	
Endosulfan I	BDL	19	
Endosulfan II	BDL	38	
Endosulfan Sulfate	BDL	38	
Endrin	BDL	38	
Endrin Aldehyde	BDL	xx	
Heptachlor	BDL	19	
Heptachlor Expoxide	BDL	19	
Toxaphene	BDL	380	
Benzyl Alcohol	BDL	xx	
4-Chloroaniline	BDL	xx	
2-Nitroaniline	BDL	xx	
3-Nitroaniline	BDL	xx	
4-Nitroaniline	BDL	xx	
2,4,5-Trichlorophenol	BDL	xx	
Chlordane, Alpha	BDL	190	
Chlordane, Gamma	BDL	190	
Endrin Ketone	BDL	38	
Methoxychlor	BDL-51000	190	
PCBs			
Arochlor-1016	BDL	190	
Arochlor-1221	BDL	190	
Arochlor-1232	BDL	190	
Arochlor-1242	BDL	190	
Arochlor-1248	BDL	190	
Arochlor-1254	BDL	390	
Arochlor-1260	BDL	390	
Other Semivolatiles			
Benzyl Butyl Phthalate	BDL	410	
Bis(2-Chloroethoxy)Methane	BDL	410	
Bis(2-Chloroethyl)Ether	BDL-370	790	
Bis(2-Chloroisopropyl)Ether	BDL	410	
Bis(2-Ethylhexyl)Phthalate	BDL-97000	410	
Benzidine	BDL	xx	
4-Bromophenyl Phenyl Ether	BDL-32000	410	
2-Chloronaphthalene	BDL	410	

TABLE 3.3-2

Summary Table
Existing Soil Quality Data, New Slip Investigation (1)

Compound	Range of Reported Concentrations (ug/kg)	Detection Limit (2) (ug/kg)	Method (3)

4-Chlorophenyl Phenyl ether	BDL-430000	410	
1,2-Dichlorobenzene	BDL	410	
1,3-Dichlorobenzene	BDL	410	
1,4-Dichlorobenzene	BDL-34000	410	
3,3'-Dichlorobenzidine	BDL	810	
Diethylphthalate	BDL-100000	410	
Dimethylphthalate	BDL-120000	410	
2,4-Dinitrotoluene	BDL-2500	410	
2,6-Dinitrotoluene	BDL-200000	410	
Di-N-Butylphthalate	BDL-230000	790	
Di-N-Octylphthalate	BDL-400000	410	
1,2-Diphenylhydrazine	BDL	xx	
Hexachlorobenzene	BDL	410	
Hexachlorobutadiene	BDL	410	
Hexachlorocyclopentadiene	BDL-5700000	410	
Hexachloroethane	BDL	410	
Isophorone	BDL	410	
Nitrobenzene	BDL	410	
N-Nitrosodiphenylamine	BDL-270000	410	
N-Nitroso-Di-N-Propylamine	BDL	790	
N-Nitrosodimethylamine	BDL	xx	
1,2,4-Trichlorobenzene	BDL	410	
VOLATILE COMPOUNDS			
Benzene	BDL-600	xx	
Bromoform	BDL	20	
Carbon Tetrachloride	BDL	20	
Chlorobenzene	BDL	20	
Chlorodibromomethane	BDL	xx	
Chloroethane	BDL	40	
2-Chloroethylvinyl Ether	BDL	xx	
Chloroform	BDL	20	
Dichlorobromomethane	BDL	20	
Dichlorodifluoromethane	BDL	xx	
1,1-Dichloroethane	BDL	20	
1,2-Dichloroethane	BDL	20	
1,1-Dichloroethylene	BDL	20	
1,2-Dichloropropane	BDL	20	
Dichloropropylene (Mixed)	BDL	xx	
Ethylbenzene	BDL-89	<15	
Methyl Bromide	BDL	xx	
Methyl Chloride	BDL	xx	

TABLE 3.3-2

Summary Table
Existing Soil Quality Data, New Slip Investigation (1)

Compound	Range of Reported Concentrations (ug/kg)	Detection Limit (2) (ug/kg)	Method (3)
Methylene Chloride	BDL	xx	
1,1,2,2-Tetrachloroethane	BDL	20	
Tetrachloroethylene	BDL	20	
Toluene	8-190	xx	
1,2-Transdichloroethylene	BDL	xx	
1,1,1-Trichloroethane	BDL	20	
1,1,2-Trichloroethane	BDL	20	
Trichloroethylene	BDL	20	
Trichlorofluoromethane	BDL	xx	
Vinyl Chloride	BDL-55	xx	
Chloromethane	BDL	40	
Bromomethane	BDL	40	
Acetone	BDL-620	44	
Carbon Disulfide	BDL	20	
2-Butanone	BDL-66	44	
Vinyl Acetate	BDL	40	
Bromodichloromethane	BDL	20	
Cis-1,3-Dichloropropene	BDL	20	
Trans-1,3-Dichloropropene	BDL	20	
4-Methyl-2-Pentanone	BDL	40	
2-Hexanone	BDL	40	
Styrene	18-36	xx	
Xylene (total)	BDL-64	25	
METALS AND CYANIDE (concentrations in ppm)			
Aluminum	1380-2930	xx	
Antimony	15.4-23.8	xx	
Arsenic	3.8-29.9	xx	
Barium	2.5	xx	
Beryllium	B	B	
Cadmium	BDL-0.5	xx	
Calcium	57800-78100	xx	
Chromium	2.1-7.0	xx	
Cobalt	BDL-10.8	4.2	
Copper	33.9-1460	xx	
Iron	4130-15100	xx	
Lead	2.3-4.4	xx	
Magnesium	28900-53200	xx	
Manganese	180-440	xx	
Mercury	BDL	0.08	
Nickel	11.7	B	
Potassium	B	131	

TABLE 3.3-2

Summary Table
Existing Soil Quality Data, New Slip Investigation (1)

Compound	Range of Reported Concentrations (ug/kg)	Detection Limit (2) (ug/kg)	Method (3)
Selenium	BDL-1.9	0.24	
Silver	BDL	0.42	
Sodium	B	B	
Thallium	BDL	0.24	
Vanadium	13.9-36.4	xx	
inc	31.9-528	xx	
Cyanide	BDL	0.54	

BDL- Below detection limit

xx- Detection limit unable to be determined

B- Blank contamination reported

(1) Canonic Environmental, Inc. 1990a.

(2) Minimum detection limits presented (where available)

(3) Information on analytical methods generally not
provided in referenced data report.

TABLE 3.3-3

Summary Table
Existing Soil Quality Data, IEPA Investigation (1)

Compound	Range of Reported Concentrations (ug/kg)	Detection Limit (2) (ug/kg)	Method (3)

SEMIVOLATILES			
PAH Compounds			
Acenaphthene	BDL-7000	XX	
Acenaphthylene	BDL-7000	XX	
Anthracene	20-29000	XX	
Benzo(a)fluoranthene	BDL-54000	XX	
Fluoranthene	200-320000	XX	
Fluorene	BDL-98000	XX	
Naphthalene	BDL-1300000	XX	
2-Methylnaphthalene	BDL-190000	XX	
Phenanthrene	140-370000	XX	
Pyrene	120-260000	XX	
Benzo(a)Anthracene (c)	200-150000	XX	
Benzo(a)Pyrene (c)	BDL-120000	XX	
Benzo(b)Fluoranthene (c)	BDL-220000	XX	
Benzo(g,h,i)Perylene (c)	BDL-47000	XX	
Benzo(k)Fluoranthene (c)	BDL-110000	XX	
Chrysene (c)	170-160000	XX	
Dibenzo(a,h)Anthracene (c)	BDL-6300	XX	
Ideno(1,2,3,c,d)Pyrene (c)	BDL-60000	XX	
Phenolic Compounds			
2-Methylphenol (O-Cresol)	BDL	XX	
4-Methylphenol (P-Cresol)	BDL-2700	XX	
2-Chlorophenol	BDL	XX	
4-Chloro-3-Methylphenol	BDL	XX	
2,4-Dichlorophenol	BDL	XX	
2,4-Dimethylphenol	BDL	XX	
2,4-Dinitrophenol	BDL	XX	
4,6-Dinitro-2-Methylphenol	BDL	XX	
2-Nitrophenol	BDL	XX	
4-Nitrophenol	BDL	XX	
Pentachlorophenol	BDL	XX	
Phenol	BDL-1500	XX	
2,4,5-Trichlorophenol	BDL	XX	
2,4,6-Trichlorophenol	BDL	XX	
Pesticides			
Aldrin	BDL	XX	
BHC, Alpha-	BDL	XX	
BHC, Beta-	BDL	XX	
BHC, Delta-	BDL	XX	

TABLE 3.3-3

Summary Table
Existing Soil Quality Data, IEPA Investigation (1)

Compound	Range of Reported Concentrations (ug/kg)	Detection Limit (2) (ug/kg)	Method (3)
BHC, Gamma- (Lindane)	BDL	xx	
4,4'-DDD (P,P'-)	NT		
4,4'-DDE (P,P'-)	NT		
4,4'-DDT (P,P'-)	NT		
Dieldrin	BDL	xx	
Endosulfan I	BDL	xx	
Endosulfan II	BDL	xx	
Endosulfan Sulfate	BDL	xx	
Endrin	BDL	xx	
Endrin Aldehyde	NT		
Heptachlor	BDL	xx	
Heptachlor Expoxide	BDL-98	xx	
Toxaphene	BDL	xx	
4-Chloroaniline	BDL	xx	
2-Nitroaniline	BDL	xx	
3-Nitroaniline	BDL	xx	
4-Nitroaniline	BDL	xx	
Chlordane, Alpha	BDL	xx	
Chlordane, Gamma	BDL	xx	
Endrin Ketone	BDL	xx	
Methoxychlor	BDL	xx	
PCBs			
Arochlor-1016	BDL	xx	
Arochlor-1221	BDL	xx	
Arochlor-1232	BDL	xx	
Arochlor-1242	BDL	xx	
Arochlor-1248	BDL	xx	
Arochlor-1254	BDL	xx	
Arochlor-1260	BDL	xx	
Other Semivolatiles			
Benzoic Acid	BDL	xx	
Benzyl Alcohol	BDL	xx	
Benzyl Butyl Phthalate	BDL-330	xx	
Bis(2-Chloroethoxy)Methane	BDL	xx	
Bis(2-Chloroethyl)Ether	BDL	xx	
Bis(2-Chloroisopropyl)Ether	BDL	xx	
Bis(2-Ethylhexyl)Phthalate	BDL-270	xx	
Benzidine	NT		
4-Bromophenyl Phenyl Ether	BDL	xx	
2-Chloronaphthalene	BDL	xx	
4-Chlorophenyl Phenyl ether	BDL	xx	

TABLE 3.3-3

Summary Table
Existing Soil Quality Data, IEPA Investigation (1)

Compound	Range of Reported Concentrations (ug/kg)	Detection Limit (2) (ug/kg)	Method (3)

1,2-Dichlorobenzene	BDL	xx	
1,3-Dichlorobenzene	BDL	xx	
1,4-Dichlorobenzene	BDL	xx	
3,3'-Dichlorobenzidine	BDL-1100	xx	
Diethylphthalate	BDL	xx	
Dimethylphthalate	BDL	xx	
2,4-Dinitrotoluene	NT		
2,6-Dinitrotoluene	BDL	xx	
Di-N-Butylphthalate	BDL-90	xx	
Di-N-Octylphthalate	BDL-170	xx	
1,2-Diphenylhydrazine	NT		
Hexachlorobenzene	BDL	xx	
Hexachlorobutadiene	BDL	xx	
Hexachlorocyclopentadiene	BDL	xx	
Hexachloroethane	BDL	xx	
Isophorone	BDL	xx	
Nitrobenzene	BDL	xx	
N-Nitrosodiphenylamine	BDL	xx	
N-Nitroso-Di-N-Propylamine	BDL	xx	
N-Nitrosodimethylamine	NT		
1,2,4-Trichlorobenzene	BDL	xx	
VOLATILE COMPOUNDS			
Benzene	BDL-1800	xx	
Bromoform	BDL	xx	
Carbon Tetrachloride	BDL	xx	
Chlorobenzene	BDL	xx	
Chlorodibromomethane	NT		
Chloroethane	NT		
2-Chloroethylvinyl Ether	NT		
Chloroform	BDL	xx	
Dichlorobromomethane	BDL	xx	
Dichlorodifluoromethane	NT		
1,1-Dichloroethane	BDL	xx	
1,2-Dichloroethane	BDL	xx	
1,1-Dichloroethylene	BDL	xx	
1,2-Dichloropropane	BDL	xx	
Dichloropropylene (Mixed)	NT		
Ethylbenzene	BDL-610	xx	
Methyl Bromide	NT		
Methyl Chloride	NT		
Methylene Chloride	0.3-350	xx	
1,1,2,2-Tetrachloroethane	BDL	xx	

TABLE 3.3-3

Summary Table
Existing Soil Quality Data, IEPA Investigation (1)

Compound	Range of Reported Concentrations (ug/kg)	Detection Limit (2) (ug/kg)	Method (3)

Tetrachloroethylene	BDL	xx	
Toluene	BDL-1900	xx	
1,2-Transdichloroethylene	NT		
1,1,1-Trichloroethane	BDL	xx	
1,1,2-Trichloroethane	BDL	xx	
Trichloroethylene	BDL	xx	
Trichlorofluoromethane	NT		
Vinyl Chloride	BDL	xx	
Chloromethane	BDL	xx	
Bromomethane	BDL	xx	
Acetone	BDL-1600	xx	
Carbon Disulfide	BDL-1.0	xx	
2-Butanone	BDL-12.0	xx	
Vinyl Acetate	BDL	xx	
Bromodichloromethane	BDL	xx	
Cis-1,3-Dichloropropene	BDL	xx	
Trans-1,3-Dichloropropene	BDL	xx	
4-Methyl-2-Pentanone	BDL	xx	
2-Hexanone	BDL	xx	
Styrene	410	xx	
Xylene (total)	0.6-8500	xx	
METALS AND CYANIDE (concentrations in ppm)			
Aluminum	1400-8240	xx	
Antimony	BDL-43.1	0.3	
Arsenic	2.3-956	xx	
Barium	8.9-150	xx	
Beryllium	1.2-11.0	xx	
Cadmium	1.0-2.9	xx	
Calcium	1250-66500	xx	
Chromium	6.0-18.0	xx	
Cobalt	B	xx	
Copper	5.0-38.0	xx	
Iron	490-23200	xx	
Lead	7.9-91.0	xx	
Magnesium	140-26500	xx	
Manganese	47-490	xx	
Mercury	BDL-58.0	xx	
Nickel	8.8-16.0	xx	
Potassium	B	xx	
Selenium	BDL-14.1	0.2	
Silver	0.1-2.0	xx	
Sodium	B	xx	

TABLE 3.3-3

Summary Table
Existing Soil Quality Data, IEPA Investigation (1)

Compound	Range of Reported Concentrations (ug/kg)	Detection Limit (2) (ug/kg)	Method (3)
Thallium	BDL	0.1	
Vanadium	9.4-26.0	xx	
Zinc	9.9-110	xx	
Cyanide	BDL-556	0.4	
Sulfate	BDL	xx	
Sulfide	BDL	xx	

BDL- Below detection limit

xx- Detection limit unable to be determined

B- Blank contamination reported

NT- Not tested

(1) IEPA, 1989.

(2) Minimum detection limit reported (where available)

(3) Information on analytical methods generally not
provided in referenced data report.

TABLE 3.4-1

INDICATOR PARAMETERS AND SUGGESTED WATER QUALITY CRITERIA

Compound	Federal Ambient Water Quality Criteria (µg/L)			State of Illinois
	Human Health Protection	Aquatic Life Protection Lowest Reported Effects Level ²		
	Ingestion of Aquatic Organisms	Acute	Chronic	General Use Water Quality Standards (µg/L) ³
VOLATILE ORGANIC COMPOUNDS				
Benzene	40 ¹	5,300	--	--
Ethyl Benzene	3,280	32,000	--	--
Toluene	424,000	17,500	--	--
POLYNUCLEAR AROMATIC HYDROCARBONS				
Acenaphthene	--	1,700	520	--
Fluoranthene	54	3,980	--	--
Potential Carcinogens				
Benzo(a)anthracene	0.031 ¹	--	--	--
Benzo(a)pyrene	0.031 ¹	--	--	--
Benzo(b)fluoranthene	0.031 ¹	--	--	--
Benzo(k)fluoranthene	0.031 ¹	--	--	--

TABLE 3.4-1 (continued)

INDICATOR PARAMETERS AND SUGGESTED WATER QUALITY CRITERIA

Compound	Federal Ambient Water Quality Criteria (µg/L)			State of Illinois
	Human Health Protection	Aquatic Life Protection Lowest Reported Effects Level ²		
	Ingestion of Aquatic Organisms	Acute	Chronic	General Use Water Quality Standards (µg/L) ³
Chrysene	0.031 ¹	--	--	--
Dibenzo(a,h)anthracene	0.031 ¹	--	--	--
Indeno(1,2,3-cd)pyrene	0.031 ¹	--	--	--
PHENOLS				
Phenol	769,000	10,200	2,560	100
INORGANICS				
Cyanide	--	44.73	7.849	25

-- No standard listed

¹Cancer risk estimated at 10^{-6} excess cancers over a 70-year lifetime of consuming an average of 6.5 grams of fish per day. The criteria for the sum of all carcinogenic PAHs is 0.031 $\mu\text{g/L}$.

²45 Federal Register 79318, November, 1980. There was not enough data to derive a numerical national water quality criteria for aquatic life protection for these chemicals.

³Substances toxic to aquatic life shall not exceed one-tenth of the 96-hour median tolerance limit (96-hr. TL_m) for native fish or essential fish food organisms.

TABLE 3.4-2

SAMPLING ACTIVITY AND SUMMARY
OF ANALYTICAL PARAMETER

	Sample Matrix	Estimated No.	Parameters
PHASE I	Soil	30	Full CLP-RAS ¹
		20	PAHs ² , TCL-VOC
		5	Acid Extractables
		3	Arsenic, Cyanide
		1	TCLP
	Groundwater	10	Full CLP-RAS
PHASE II	Soil	140	PAHs, BTEX
		28	Acid Extractables
		10	Arsenic, Cyanide, Corrosivity, Reactivity
		3	TCLP
		3-6	Soil Characteristic Testing ⁶
	Groundwater	22 ⁴	PAHs, BTEX
		22	Acid Extractables
		6	Water quality parameters ⁵

¹Includes both organic TCL and inorganic TAL compounds

²Polynuclear aromatic hydrocarbon compounds

³Benzene, toluene, ethyl benzene, xylene

⁴Selected samples will be analyzed for PAHs using the low level GC/MS method based on results of preliminary PAH analyses (Appendix B)

⁵Total suspended solids, oil and grease, BOD, COD

⁶Soil classification, grain size, bulk density, specific gravity, vertical permeability, Atterberg limits, TOC, BTU, flashpoint

TABLE 3.4-3

PAH AND ACID EXTRACTABLE COMPOUNDS

<u>Polynuclear Aromatic Hydrocarbon Compounds (PAH's)</u>	<u>Acid Extractable Compounds (Phenols)</u>
Naphthalene	Phenol
2-Methylnaphthalene	2-Chlorophenol
Acenaphthylene	2-Methylphenol
Acenaphthene	4-Methylphenol
Dibenzofuran	2-Nitrophenol
Fluorene	2,4-Dimethylphenol
Phenanthrene	4-Chloro-3-methylphenol
Anthracene	2,4,6-Trichlorophenol
Fluoranthene	2,4,5-Trichlorophenol
Pyrene	2,4-Dinitrophenol
Benzo(a)anthracene	4-Nitrophenol
Chrysene	4,6-Dinitro-2-methylphenol
Benzo(b&k)fluoranthene	Pentachlorophenol
Benzo(a)pyrene	
Indeno(1,2,3-cd)pyrene	
Dibenzo(a,h)anthracene	
Benzo(g,h,i)perylene	

Analysis of the above compounds will be performed following procedures in CLP-SOW OLM01.1.

TABLE 3.4-4
CLP ANALYTICAL PARAMETERS AND QUANTITATION LIMITS

TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

Volatiles	CAS Number	Quantitation Limits*			On Column (ng)
		Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	
1. Chloromethane	74-87-3	10	10	1200	(50)
2. Bromomethane	74-83-9	10	10	1200	(50)
3. Vinyl Chloride	75-01-4	10	10	1200	(50)
4. Chloroethane	75-00-3	10	10	1200	(50)
5. Methylene Chloride	75-09-2	10	10	1200	(50)
6. Acetone	67-64-1	10	10	1200	(50)
7. Carbon Disulfide	75-15-0	10	10	1200	(50)
8. 1,1-Dichloroethene	75-35-4	10	10	1200	(50)
9. 1,1-Dichloroethane	75-34-3	10	10	1200	(50)
10. 1,2-Dichloroethene (total)	540-59-0	10	10	1200	(50)
11. Chloroform	67-66-3	10	10	1200	(50)
12. 1,2-Dichloroethane	107-06-2	10	10	1200	(50)
13. 2-Butanone	78-93-3	10	10	1200	(50)
14. 1,1,1-Trichloroethane	71-55-6	10	10	1200	(50)
15. Carbon Tetrachloride	56-23-5	10	10	1200	(50)
16. Bromodichloromethane	75-27-4	10	10	1200	(50)
17. 1,2-Dichloropropane	78-87-5	10	10	1200	(50)
18. cis-1,3-Dichloropropene	10061-01-5	10	10	1200	(50)
19. Trichloroethene	79-01-6	10	10	1200	(50)
20. Dibromochloromethane	124-48-1	10	10	1200	(50)
21. 1,1,2-Trichloroethane	79-00-5	10	10	1200	(50)
22. Benzene	71-43-2	10	10	1200	(50)
23. trans-1,3-Dichloropropene	10061-02-6	10	10	1200	(50)
24. Bromoform	75-25-2	10	10	1200	(50)
25. 4-Methyl-2-pentanone	108-10-1	10	10	1200	(50)
26. 2-Hexanone	591-78-6	10	10	1200	(50)
27. Tetrachloroethene	127-18-4	10	10	1200	(50)
28. Toluene	108-88-3	10	10	1200	(50)
29. 1,1,2,2-Tetrachloroethane	79-34-5	10	10	1200	(50)
30. Chlorobenzene	108-90-7	10	10	1200	(50)
31. Ethyl Benzene	100-41-4	10	10	1200	(50)
32. Styrene	100-42-5	10	10	1200	(50)
33. Xylenes (Total)	1330-20-7	10	10	1200	(50)

* Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

TABLE 3.4-4 (continued)

CLP ANALYTICAL PARAMETERS AND QUANTITATION LIMITS
TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

Semivolatiles	CAS Number	Quantitation Limits*			
		Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)
34. Phenol	108-95-2	10	330	10000	(20)
35. bis(2-Chloroethyl) ether	111-44-4	10	330	10000	(20)
36. 2-Chlorophenol	95-57-8	10	330	10000	(20)
37. 1,3-Dichlorobenzene	541-73-1	10	330	10000	(20)
38. 1,4-Dichlorobenzene	106-46-7	10	330	10000	(20)
39. 1,2-Dichlorobenzene	95-50-1	10	330	10000	(20)
40. 2-Methylphenol	95-48-7	10	330	10000	(20)
41. 2,2'-oxybis (1-Chloropropane) #	108-60-1	10	330	10000	(20)
42. 4-Methylphenol	106-44-5	10	330	10000	(20)
43. N-Nitroso-di-n- propylamine	621-64-7	10	330	10000	(20)
44. Hexachloroethane	67-72-1	10	330	10000	(20)
45. Nitrobenzene	98-95-3	10	330	10000	(20)
46. Isophorone	78-59-1	10	330	10000	(20)
47. 2-Nitrophenol	88-75-5	10	330	10000	(20)
48. 2,4-Dimethylphenol	105-67-9	10	330	10000	(20)
49. bis(2-Chloroethoxy) methane	111-91-1	10	330	10000	(20)
50. 2,4-Dichlorophenol	120-83-2	10	330	10000	(20)
51. 1,2,4-Trichlorobenzene	120-82-1	10	330	10000	(20)
52. Naphthalene	91-20-3	10	330	10000	(20)
53. 4-Chloroaniline	106-47-8	10	330	10000	(20)
54. Hexachlorobutadiene	87-68-3	10	330	10000	(20)
55. 4-Chloro-3-methylphenol	59-50-7	10	330	10000	(20)
56. 2-Methylnaphthalene	91-57-6	10	330	10000	(20)
57. Hexachlorocyclopentadiene	77-47-4	10	330	10000	(20)
58. 2,4,6-Trichlorophenol	88-06-2	10	330	10000	(20)
59. 2,4,5-Trichlorophenol	95-95-4	25	800	25000	(50)
60. 2-Chloronaphthalene	91-58-7	10	330	10000	(20)
61. 2-Nitroaniline	88-74-4	25	800	25000	(50)
62. Dimethylphthalate	131-11-3	10	330	10000	(20)
63. Acenaphthylene	208-96-8	10	330	10000	(20)
64. 2,6-Dinitrotoluene	606-20-2	10	330	10000	(20)
65. 3-Nitroaniline	99-09-2	25	800	25000	(50)
66. Acenaphthene	83-32-9	10	330	10000	(20)
67. 2,4-Dinitrophenol	51-28-5	25	800	25000	(50)
68. 4-Nitrophenol	100-02-7	25	800	25000	(50)

Previously known by the name bis(2-Chloroisopropyl) ether

TABLE 3.4-4 (continued)

CLP ANALYTICAL PARAMETERS AND QUANTITATION LIMITS

(continued)		Quantitation Limits*			
Semivolatiles	CAS Number	Low		Med.	On
		Water	Soil	Soil	Column
		ug/L	ug/Kg	ug/Kg	(ng)
69. Dibenzofuran	132-64-9	10	330	10000	(20)
70. 2,4-Dinitrotoluene	121-14-2	10	330	10000	(20)
71. Diethylphthalate	84-66-2	10	330	10000	(20)
72. 4-Chlorophenyl-phenyl ether	7005-72-3	10	330	10000	(20)
73. Fluorene	86-73-7	10	330	10000	(20)
74. 4-Nitroaniline	100-01-6	25	800	25000	(50)
75. 4,6-Dinitro-2-methylphenol	534-52-1	25	800	25000	(50)
76. N-nitrosodiphenylamine	86-30-6	10	330	10000	(20)
77. 4-Bromophenyl-phenylether	101-55-3	10	330	10000	(20)
78. Hexachlorobenzene	118-74-1	10	330	10000	(20)
79. Pentachlorophenol	87-86-5	25	800	25000	(50)
80. Phenanthrene	85-01-8	10	330	10000	(20)
81. Anthracene	120-12-7	10	330	10000	(20)
82. Carbazole	86-74-8	10	330	10000	(20)
83. Di-n-butylphthalate	84-74-2	10	330	10000	(20)
84. Fluoranthene	206-44-0	10	330	10000	(20)
85. Pyrene	129-00-0	10	330	10000	(20)
86. Butylbenzylphthalate	85-68-7	10	330	10000	(20)
87. 3,3'-Dichlorobenzidine	91-94-1	10	330	10000	(20)
88. Benzo(a)anthracene	56-55-3	10	330	10000	(20)
89. Chrysene	218-01-9	10	330	10000	(20)
90. bis(2-Ethylhexyl)phthalate	117-81-7	10	330	10000	(20)
91. Di-n-octylphthalate	117-84-0	10	330	10000	(20)
92. Benzo(b)fluoranthene	205-99-2	10	330	10000	(20)
93. Benzo(k)fluoranthene	207-08-9	10	330	10000	(20)
94. Benzo(a)pyrene	50-32-8	10	330	10000	(20)
95. Indeno(1,2,3-cd)pyrene	193-39-5	10	330	10000	(20)
96. Dibenz(a,h)anthracene	53-70-3	10	330	10000	(20)
97. Benzo(g,h,i)perylene	191-24-2	10	330	10000	(20)

* Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

TABLE 3.4-4 (continued)

CLP ANALYTICAL PARAMETERS AND QUANTITATION LIMITS

TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)

Pesticides/Aroclors	CAS Number	Quantitation Limits*		
		Water ug/L	Soil ug/Kg	On Column (pg)
98. alpha-BHC	319-84-6	0.05	1.7	5
99. beta-BHC	319-85-7	0.05	1.7	5
100. delta-BHC	319-86-8	0.05	1.7	5
101. gamma-BHC (Lindane)	58-89-9	0.05	1.7	5
102. Heptachlor	76-44-8	0.05	1.7	5
103. Aldrin	309-00-2	0.05	1.7	5
104. Heptachlor epoxide	1024-57-3	0.05	1.7	5
105. Endosulfan I	959-98-8	0.05	1.7	5
106. Dieldrin	60-57-1	0.10	3.3	10
107. 4,4'-DDE	72-55-9	0.10	3.3	10
108. Endrin	72-20-8	0.10	3.3	10
109. Endosulfan II	33213-65-9	0.10	3.3	10
110. 4,4'-DDD	72-54-8	0.10	3.3	10
111. Endosulfan sulfate	1031-07-8	0.10	3.3	10
112. 4,4'-DDT	50-29-3	0.10	3.3	10
113. Methoxychlor	72-43-5	0.50	17.0	50
114. Endrin ketone	53494-70-5	0.10	3.3	10
115. Endrin aldehyde	7421-36-3	0.10	3.3	10
116. alpha-Chlordane	5103-71-9	0.05	1.7	5
117. gamma-Chlordane	5103-74-2	0.05	1.7	5
118. Toxaphene	8001-35-2	5.0	170.0	500
119. Aroclor-1016	12674-11-2	1.0	33.0	100
120. Aroclor-1221	11104-28-2	2.0	67.0	200
121. Aroclor-1232	11141-16-5	1.0	33.0	100
122. Aroclor-1242	53469-21-9	1.0	33.0	100
123. Aroclor-1248	12672-29-6	1.0	33.0	100
124. Aroclor-1254	11097-69-1	1.0	33.0	100
125. Aroclor-1260	11096-82-5	1.0	33.0	100

* Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of Pesticides/Aroclors.

(TABLE 3.6-1 (continued) (

SAMPLE AND ANALYSIS PROGRAM

PHASE II

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative Sample			Field Quality Control Samples						MS/MSD			Matrix Total
						Field Duplicate			Field Blank						
			No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	
Soil	Screening for Total Organic Vapors	PAHs	140	1	140	14	1	14	-	-	-	7	1	7	154
		Acid Extractables	28	1	28	3	1	3	-	-	-	2	1	2	31
	Field Soil Classification	BETX	140	1	140	14	1	14	-	-	-	7	1	7	154
		Arsenic & Cyanide,	10	1	10	1	1	1	-	-	-	-	-	-	11
		Corrosivity & Reactivity	10	1	10	1	1	1	-	-	-	-	-	-	11
	Field pH														
	Field Screening for PAHs	Grain Size Distribution	6	1	6	-	-	-	-	-	-	-	-	-	6
		Atterberg Limits	3	1	3	-	-	-	-	-	-	-	-	-	3
		Porosity	6	1	6	-	-	-	-	-	-	-	-	-	6
		Total Organic Carbon	6	1	6	-	-	-	-	-	-	-	-	-	6
		Vertical Permeability	3	1	3	-	-	-	-	-	-	-	-	-	3
		TCLP	3	1	3	-	-	-	-	-	-	-	-	-	3
		Gross Heating Value	3	1	3	-	-	-	-	-	-	-	-	-	3
		Flashpoint	3	1	3	-	-	-	-	-	-	-	-	-	3
Groundwater	pH, Temperature Specific Conductance eH, Dissolved Oxygen	PAHs (Selected samples for low level)	22	1	22	3	1	3	3	1	3	2	1	2	28
	Slug Test	Acid Extractables	22	1	22	3	1	3	3	1	3	2	1	2	28
	Pumping Test	BETX	22	1	22	3	1	3	3	1	3	2	1	2	28
		Total Suspended Solids	6	1	6	1	1	1	-	-	-	-	-	-	10
	Slug Test	Oil and Grease	6	1	6	1	1	1	-	-	-	-	-	-	10
	BOD/COD	6	1	6	1	1	1	-	-	-	-	-	-	10	

1. The field quality control samples also include trip blank, which is required for VOA water and air samples. One trip blank, which consists of two 40-ml glass vials for water samples and one blank cartridge for air samples, is shipped with each shipping cooler of VOA samples.
2. Matrix spike/matrix spike duplicate (MS/MSD) is required for organic analysis. Water samples designated for MS/MSD analysis will be collected with extra sample volumes, at a frequency of one per group of 20 or fewer investigative samples. Triple the normal sample volumes will be collected for VOAs, and double the normal sample volumes will be collected for extractable organics, pesticides, and PCBs.
3. For inorganic analysis, no extra sample volume is required for inorganic MS/MSD samples.
4. The number of samples to be collected for MS/MSD are not included in the matrix total. The number of trip blank samples is also excluded from the matrix total.
5. All samples for metals will be filtered in the field.

TABLE 3.7-1

PROJECTED SCHEDULE - WCP SITE RI/FS

ACTIVITY	START	TASKS	TASK DURATION (WEEKS)	CUMULATIVE DURATION ⁽¹⁾ (WEEKS)
Phase I Field Investigation	Work Plan Approved	Investigation Support	7	7
		Test Trenching	4	9
		Surficial Soil/Background Sampling	4	9
		Monitoring Wells/Soil Borings	4	13
		Groundwater Sampling/Slug Tests	2	15
		Ecological Survey	2	19
		Sample Analysis/Validation	11	19
		Data Evaluation/Modeling	13	21
		Phase I Tech Memo	9	26
		Revised ARARs/PRG Tech Memo	5	27
		EPA Review	4	30
Phase II Field Investigation	Phase I Tech Memo Approved	Investigation Support	9	39
		Soil Borings	5	39
		Monitoring Wells	4	43
		Pumping Test	1	43
		Groundwater Sampling (1)	1	43
		Sample Analysis/Validation (1)	11	50
		Groundwater Sampling (2)	1	50
		Sample Analysis/Validation (2)	4	54
		Data Evaluation	22	60
		Preliminary Characterization Summary	5	60
		EPA Review	2	62

⁽¹⁾Accounts for concurrent tasks.

TABLE 3.7-1 (continued)

PROJECTED SCHEDULE - WCP SITE RI/FS

ACTIVITY	START	TASKS	TASK DURATION (WEEKS)	CUMULATIVE DURATION ⁽¹⁾ (WEEKS)
RI Report/RA Development and Screening	Preliminary Characterization Summary Approved	Prepare Draft RI Report	12	71
		Prepare Tech Memo on Technologies and Screening Process	12	71
		EPA Review	2	73
		Revisions to Draft RI	4	77
		Prepare Screened Alternatives and Proposed ARARS Tech Memo	9	79
		EPA Approval; Risk Assessment	2;68	79
Alternatives Summary and Evaluation/FS Report	EPA Risk Assessment Completed	PRP Review of Risk Assessment	4	79
		EPA Response	4	79
		Prepare Tech Memo on Alternatives Array Summary	5	84
		Prepare Tech Memo on Comparative Analysis of Alternatives	8	92
		Prepare Draft FS Report	10	97
		EPA Review	2	99
		Revisions to Draft FS/ Submittal of Final FS	5	104
PROJECT TOTAL:				104 weeks (24 months)

⁽¹⁾Accounts for concurrent tasks.

**TABLE 3.7-2
ESTIMATED PROJECT SCHEDULE**

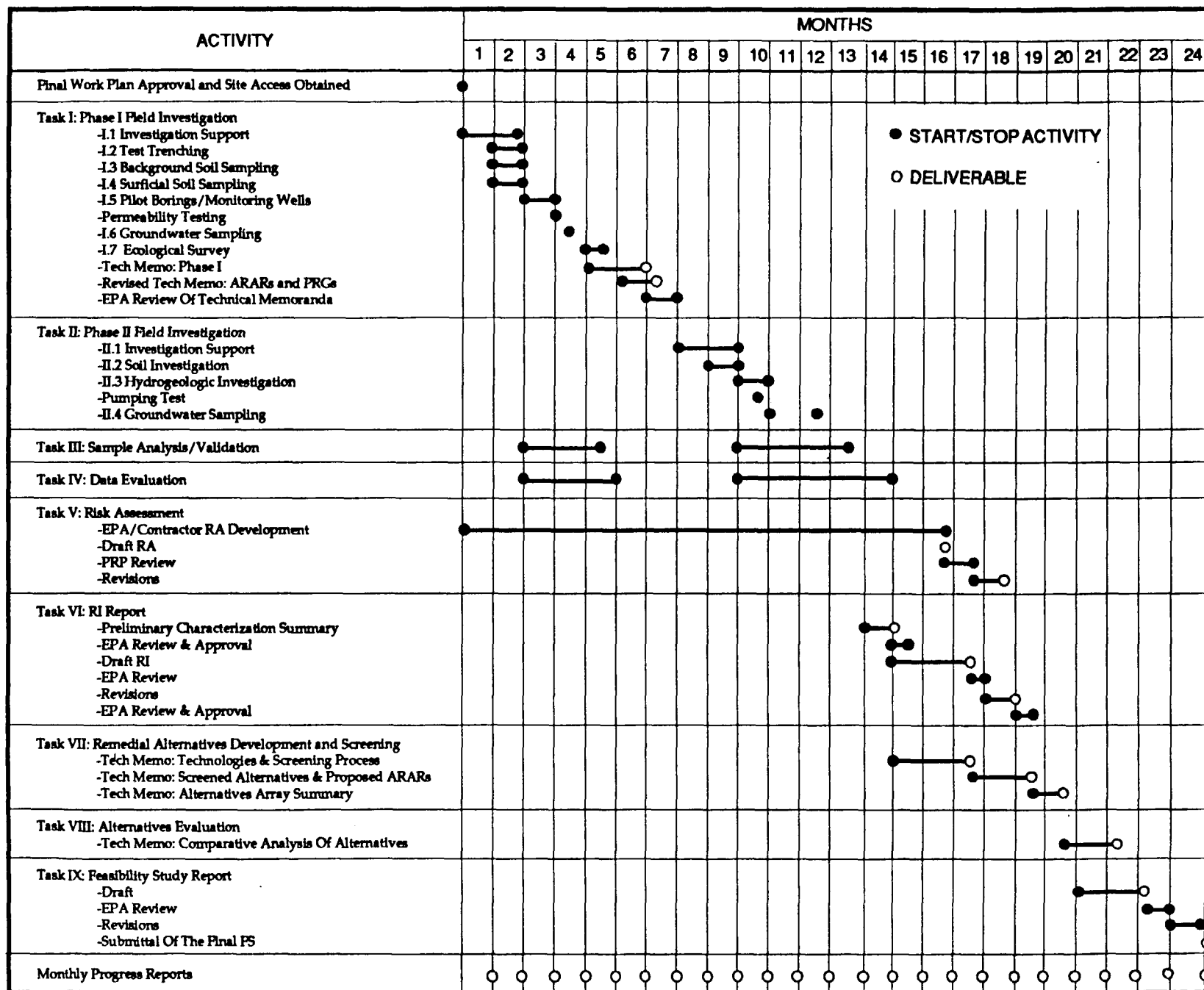


TABLE 6.6-1

SAMPLE CONTAINERS, PRESERVATION AND HOLDING TIMES

Groundwater Parameter	Container	Preservation	Maximum Holding Time	Minimum Volume
Semivolatiles: PAHs (including low level), Acid Extractables	Amber glass, teflon-lined cap	Cool, 4°C	5 days to extraction; 40 days after extraction	2,000 ml
Cyanide	High density polyethylene or glass	Cool, 4°C, NaOH to pH>12	12 days	1,000 ml
Metals (except Hg)	High density polyethylene or glass	HNO ₃ to pH<2	6 months	1,000 ML
Mercury	High density polyethylene or glass	HNO ₃ to pH<2	26 days	500 ml
Oil and Grease	Glass	Cool, 4°C, H ₂ SO ₄ to pH<2	28 days	2,000 ml
Total Suspended Solids	High density polyethylene or glass	Cool, 4°C	7 days	200 ml
Pesticides/PCBs	Amber glass, teflon-lined cap	Cool, 4°C	5 days extraction; 40 days after extraction	2,000 ml
Chemical Oxygen Demand	High density polyethylene or glass	Cool, 4°C, H ₂ SO ₄ to pH<2	26 days	100 ml
Biochemical Oxygen Demand	High density polyethylene or glass	Cool, 4°C	48 hours	500 ml
Volatile Organic Compounds	Glass, teflon-lined septum	Cool, 4°C, HCl to pH<2	10 days	40 mlx2

- d. Sample type? (Y/N) _____
- e. Identification of well? (Y/N) _____
- f. Number of containers? (Y/N) _____
- g. Parameters requested? (Y/N) _____
- h. Signatures of persons involved in the chain-of-possession? (Y/N) _____
- i. Inclusive dates of possession? (Y/N) _____

VI. REVIEW OF QUALITY ASSURANCE/QUALITY CONTROL

- A. Is the validity and reliability of the laboratory and field generated data ensured by a QA/QC program? (Y/N) _____
- B. Does the QA/QC program include:
 - 1. Documentation of any deviations from approved procedures? (Y/N) _____
 - 2. Collection and analysis of trip blanks and equipment blanks? (Y/N) _____
 - 3. Collection and analysis of blind duplicate samples? (Y/N) _____

TABLE 6.6-1 (continued)

SAMPLE CONTAINERS, PRESERVATION AND HOLDING TIMES

Soil Parameter	Container	Preservation	Maximum Holding Time	Minimum Volume
Semivolatiles: PAHs, Acid Extractables, Pesticides/PCBs	Glass, teflon-lined cap	Cool, 4°C	10 days to extraction; 40 days after extraction	8 oz.
Cyanide	High density polyethylene or glass	Cool, 4°C	12 days	8 oz.
Metals (except mercury)	High density polyethylene or glass	Cool, 4°C	180 days	8 oz.
Mercury	High density polyethylene or glass	Cool, 4°C	26 days	8 oz.
Volatiles	Glass, teflon	Cool, 4°C	10 days	2 - 2 oz. jars
TCLP	Glass	Cool, 4°C	See table below	32 oz.
TOC	Glass	Cool 4°C	28 days	8 oz.
Flashpoint	Glass	Cool 4°C	not defined	8 oz.
Reactivity/Corrosivity	Glass	Cool 4°C	as soon as possible	8 oz.

TCLP SAMPLE MAXIMUM HOLDING TIMES (days)

	From Field Collection to TCLP Extraction	From TCLP Extraction to Preparative Extraction	From Preparative Extraction to Determinative Analysis	Total Elapsed Time
Volatiles	14	NA	14	28
Semivolatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360

TABLE 9.3-1

SOIL CHARACTERIZATION PARAMETERS AND PROCEDURES

Parameter	Method of Analysis
Soil Classification	ASTM D2487 or ASTM D2488
Grain Size Distribution	ASTM D422
Bulk Density/Porosity	ASTM D653
Specific Gravity	ASTM D653
Vertical Permeability Coarse Grain Soil Fine Grain Soil	ASTM D2434 - Constant Head "Flexible Wall" COE 9100
Atterberg Limits	ASTM D4318
Total Organic Carbon	EPA 415.1 (modified)
BTU - Gross Heating Value	ASTM D240-76
Flashpoint	EPA SW 846 Chapter 7 (7.1)
TCLP	EPA 40 CFR Parts 261, 264, 265, 268, 271, and 302
Corrosivity	EPA SW 846 9045
Reactivity	EPA SW 846 9010/9030, Chapter 7

TABLE 11.3-1

DATA SUMMARY SPREADSHEET

(concentrations in ug/kg)

	S83504	S83506
	-----	-----
	12/17/90	12/17/90
2,3-Benzofuran	28000 j	<400
2,3-Dihydroindene	9800 j	<400
Indene	150000	120 j
Naphthalene	1200000	2400
Benzo(b)thiophene	32000 j	65 j
Quinoline	37000 j	200 j
Isoquinoline	11000 j	50 j
2-Methylnaphthalene	310000	880
Indole	<79000	<400
1-Methylnaphthalene	190000	560
Biphenyl	37000 j	130 j
Acenaphthylene	210000	930
Acenaphthene	36000 j	130 j
Dibenzofuran	110000	430
Fluorene	180000	830
Dibenzothiophene	23000 j	140 j
Phenanthrene	470000	2500
Anthracene	160000	830
Acridine	<79000	<400
Phenanthridine	16000 j	96 j
Carbazole	44000 j	250 j
Fluoranthene	270000	1300
Pyrene	210000	1100
Benzo(a)anthracene	88000	430
Chrysene	76000 jc	370 jc
Triphenylene	76000 jc	370 jc
Benzo(b)fluoranthene	87000 c	410 c
Benzo(k)fluoranthene	87000 c	410 c
7,12-Dimethylbenz(a)anthracene	<79000	<400
Benzo(e)pyrene	19000 j	99 j
Benzo(a)pyrene	52000 j	280 j
Perylene	11000 j	47 j
3-Methylcholanthrene	<79000	<400
Indeno(1,2,3,cd)pyrene	35000 j	120 j
Dibenzo(ah)anthracene	<79000	<400
Benzo(ghi)perylene	31000 j	130 j
Sum of Carcinogens	340000	1600
Sum of Non-Carcinogens	3200000	11000

j Reported value is less than quantitation limit.
c Compounds coelute.

Figures

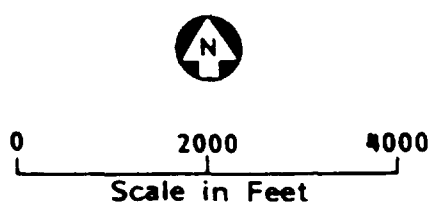
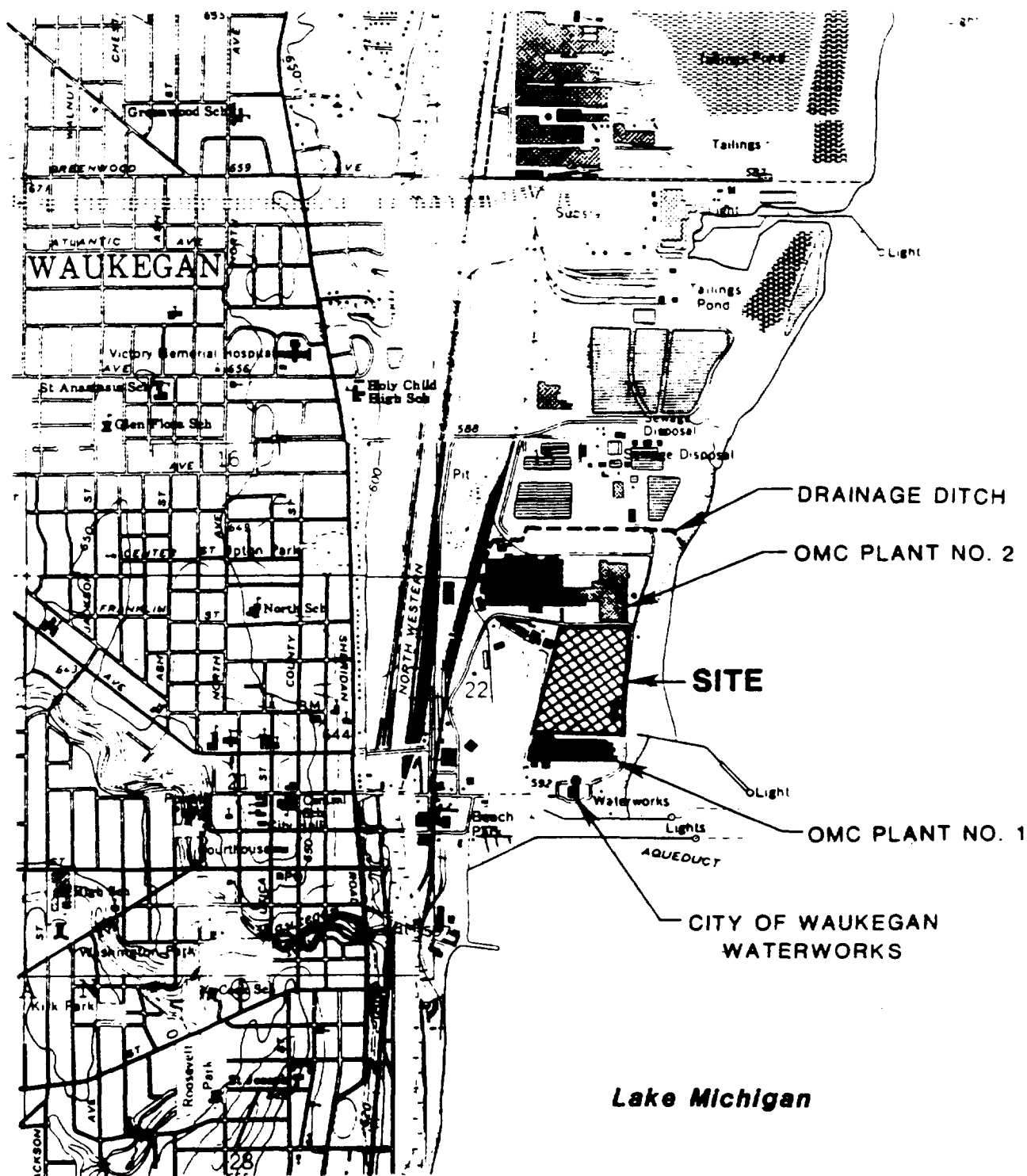


Figure 3.2-1
Site Location Map

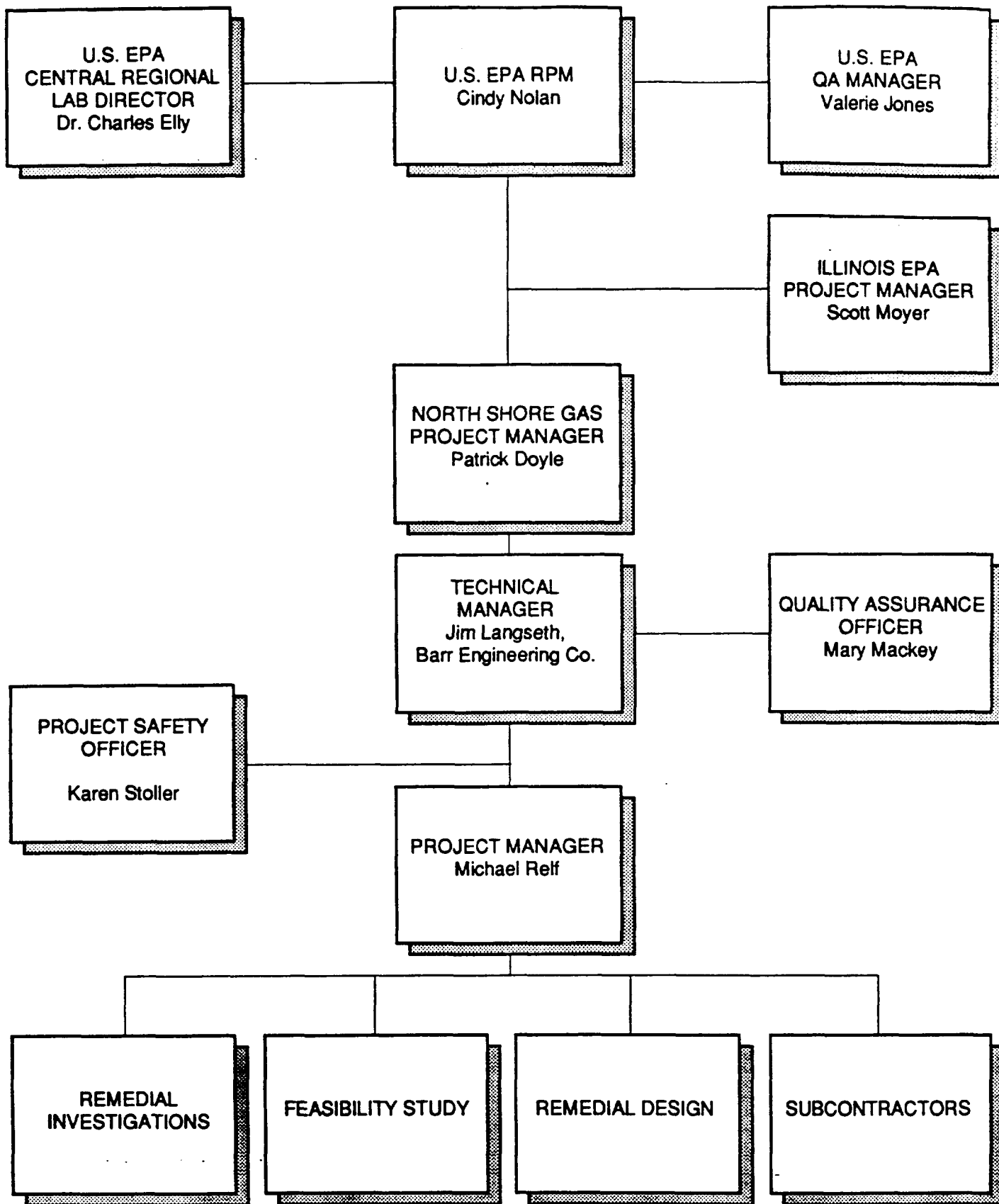
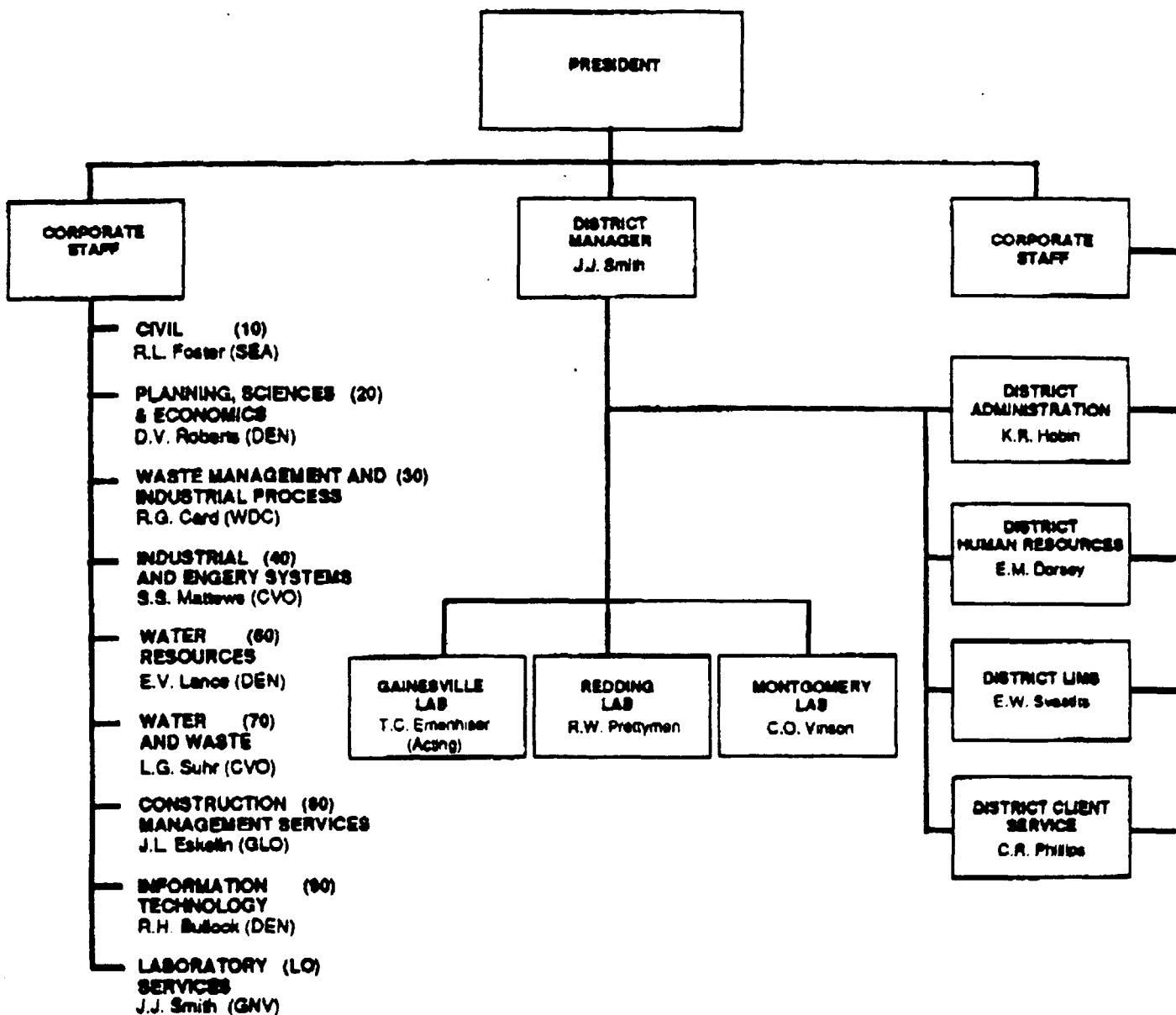


Figure 4.1-1

PROJECT ORGANIZATION CHART
WCP SITE RI/FS

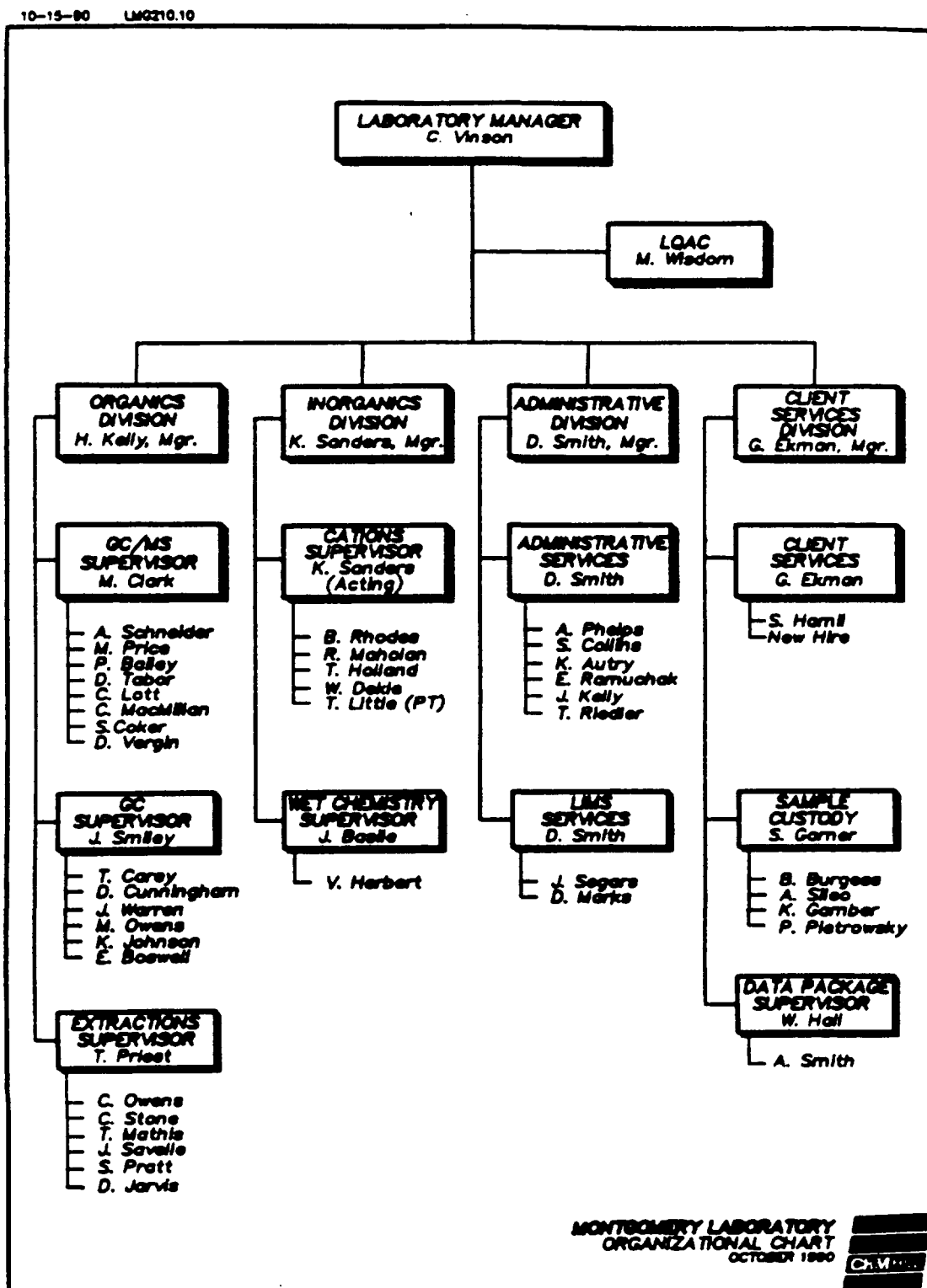
Figure 4.1-2

LABORATORY ORGANIZATIONAL CHART



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Revision No. 1
Date: October 1990
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Figure 4.1-3
ORGANIZATION CHART



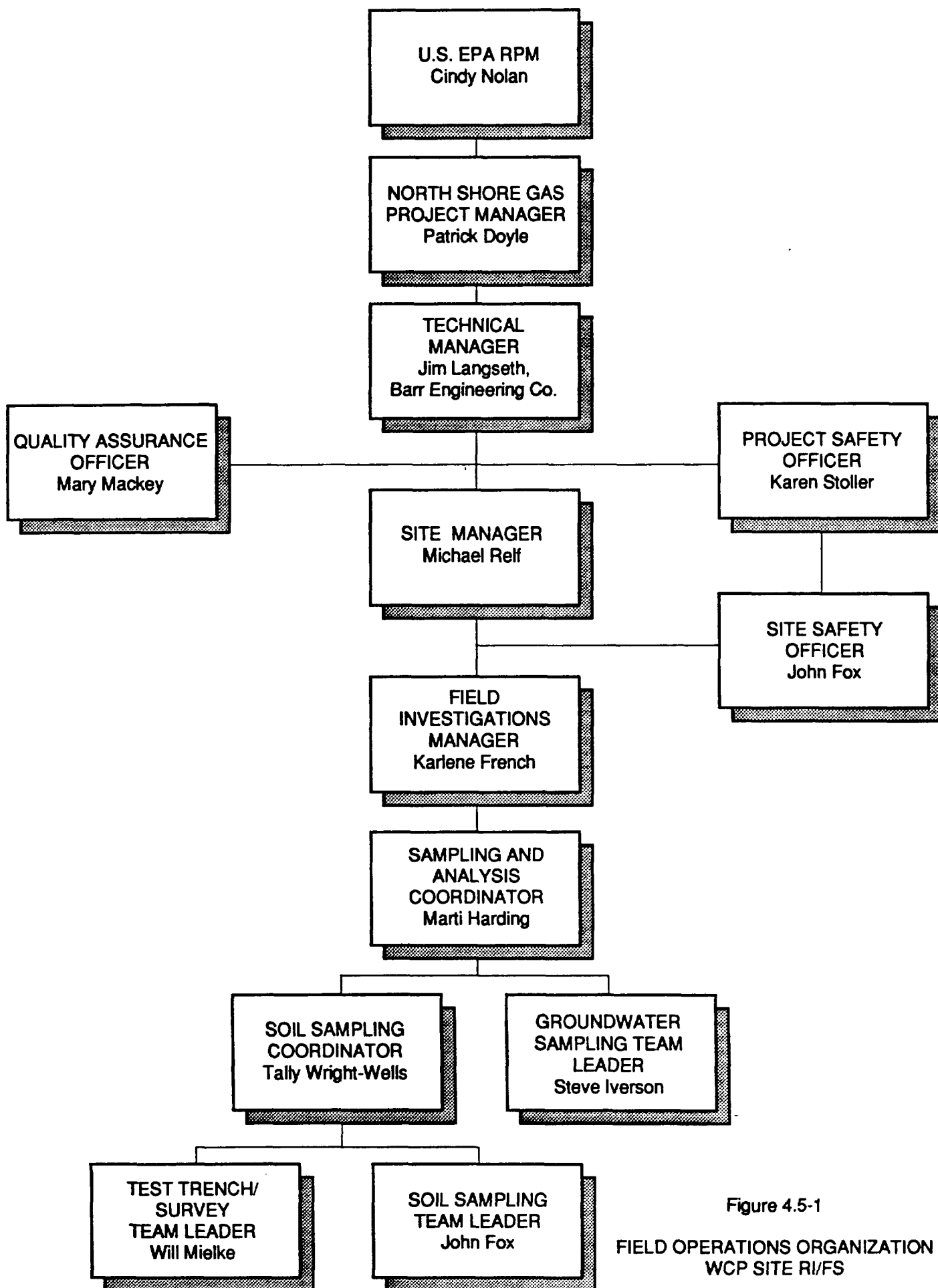


Figure 4.5-1

FIELD OPERATIONS ORGANIZATION
WCP SITE RI/FS

Figure 7.2-2

SAMPLE LABEL


	PH. (205) 271-1444
	Montgomery Laboratory 2567 Fairlane Drive Montgomery, Alabama 36116
Client _____	
Sample No. _____	
Location _____	
Analysis _____	
Preservative _____	
Date _____ By _____	

Figure 7.2-3
CUSTODY SEAL

INITIALS _____ DATE _____	EVIDENCE DO NOT OPEN	INITIALS _____ DATE _____	EVIDENCE DO NOT OPEN
	EVIDENCE DO NOT OPEN		EVIDENCE DO NOT OPEN

TABLE 3.6-1

SAMPLE AND ANALYSIS PROGRAM

PHASE I

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative Sample			Field Quality Control Samples						MS/MSD			Matrix Total	
						Field Duplicate			Field Blank							
			No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total		
Soil	Screening for Total Organic Vapors	CLP TCL Volatile Organics	50	1	50	5	1	5	-	-	-	1	1	1	55	
		CLP TCL Extractables	3027	1	27	3	1	3	-	-	-	1	1	1	3330	
		CLP TCL Pesticides/PCBs	3027	1	27	3	1	3	-	-	-	1	1	1	3330	
	Field Soil Classification	CLP TAL Metals/Cyanide	3027	1	27	3	1	3	-	-	-	1	1	1	3330	
		Field Screening for PAHs	PAHS	2025	1	23	3	1	3	-	-	-	2	1	2	2326
	Field Screening for PAHs	Acid Extractables	5	1	5	1	1	1	-	-	-	1	1	1	6	
		Arsenic and Cyanide	3	1	3	1	1	1	-	-	-	-	-	-	4	
		TCLP	1	1	1	1	1	1	-	-	-	-	-	-	2	
		Groundwater	pH, Temperature Specific Conductance	CLP TCL Volatile Organics	10	1	10	1	1	1	1	1	1	1	1	1
	CLP TCL Extractables			10	1	10	1	1	1	1	1	1	1	1	1	12
CLP TCL Pesticides/PCBs	10			1	10	1	1	1	1	1	1	1	1	1	12	
CLP TAL Metals/Cyanide	10			1	10	1	1	1	1	1	1	1	1	1	12	

1. The field quality control samples also include trip blank, which is required for VOA water and air samples. One trip blank, which consists of two 40-ml glass vials for water samples and one blank cartridge for air samples, is shipped with each shipping cooler of VOA samples.
2. Matrix spike/matrix spike duplicate (MS/MSD) is required for organic analysis. Samples designated for MS/MSD analysis will be collected with extra sample volumes, at a frequency of one per group of 20 or fewer investigative samples. Triple the normal sample volumes will be collected for VOAs, and double the normal sample volumes will be collected for extractable organics, pesticides, and PCBs.
3. For inorganic analysis, no extra sample volume is required for inorganic MS/MSD samples.
4. The number of samples to be collected for MS/MSD are not included in the matrix total. The number of trip blank samples is also excluded from the matrix total.
5. All samples for metals will be filtered in the field.

TABLE 3.5-4
DATA QUALITY OBJECTIVES

<u>Sample Type</u>	<u>Field Parameters</u>	<u>Laboratory Parameters</u>	<u>Analytical Level</u>
Soil	Field Screening (OVA)	--	I
	Soil Classification	--	I
		PAHs	IV
		TCL-VOCs	IV
		VOCs (benzene, toluene, xylene, ethyl benzene)	III
		Acid Extractables	IV
		Arsenic, Cyanide, Reactivity, Corrosivity	IV III
		Full CLP-RAS: TCL Volatiles	IV
		TCL Semivolatiles	IV
		TCL Pesticides/PCBs	IV
		TAL Metals and Cyanide	IV
		Soil Characteristic Testing	III
		TCLP	III
Groundwater	pH, Conductivity, Temperature	--	I
		Full CLP-RAS: TCL Volatiles	IV
		TCL Semivolatiles	IV
		TCL Pesticides/PCBs	IV
		TAL Metal and Cyanide	IV
		PAHs	IV
		VOCs (benzene, toluene, xylene, ethyl benzene)	III
		Acid Extractables	IV
		General Chemistry	III
		Low Level PAH Compounds	V

TABLE 3.5-3 (continued)

Sampling Activity	Objective	Estimated No. of Samples	Analytical Parameters	Rationale for Sample Selection
Groundwater Sampling	Refine groundwater quality characterization	22	PAHs, BETX, Phenols	1st Round from Phase II monitoring wells; 2nd Round from all monitoring wells; selected samples for low-level PAH analysis based on Phase I results.
	Assess potential treatability alternatives	6	BOD/COD, oil & grease, total suspended solids	1 sample from each well showing relatively high chemical constituent concentrations.

TABLE 3.5-3

INTENDED DATA USAGE AND SAMPLE RATIONALE PHASE II

Sampling Activity	Objective	Estimated No. of Samples	Analytical Parameters	Rationale for Sample Selection
Soils Investigation	Stratigraphic characterization and qualitative identification of PAH-contaminated soil for selection of samples for analyses	480	Field soil classification, visual examination, field oil sheen test, odor observations, field headspace organic vapor screening, field pH	Split-barrel samples collected from borings at 2½-foot intervals
	Assess vertical and horizontal extent of key chemical constituents	102	PAHs, BETX	One near-surface sample from each boring; one sample from sand/till contact at each boring; additional samples based on visual contamination, stratigraphy
	As above, plus assess levels of phenols and possible correlation with PAHs	28	PAHs, BETX, Phenols	Approximately 20% of samples analyzed for PAHs; assess different levels of visual contamination
	Assess vertical and horizontal extent of key chemical constituents and characteristics associated with thionizer process	10	PAHs, BETX, Arsenic, Cyanide, Corrosivity, Reactivity	From borings near Thionizer Building/Sulfur Pile
	Determine basic soil characteristic data	6	Grain size distribution, porosity	3 samples from till, 3 from sand to provide areally representative samples
	Determine basic soil characteristic data	3	Atterberg Limits	1 sample of till from each of 3 borings to provide areally representative samples
	Assess adsorptive nature of sand and till units	6	Total organic carbon	3 samples from till, 3 from sand to provide areally representative samples
	Assess vertical permeability of till	3	Vertical permeability	1 sample from each of 3 borings to provide areally representative samples
	Assess potential treatability alternatives	3	TCLP, gross heating value, flashpoint	3 samples from different areas of soils identified as containing coal tar

TABLE 3.5-2
INTENDED DATA USAGE AND SAMPLE RATIONALE PHASE I

Sampling Activity	Objective	Estimated No. of Samples	Analytical Parameters	Rationale for Sample Selection
Preliminary Source Area Characterization	Qualitatively identify PAH-contaminated soil for selection of sample locations and Phase II boring locations	140	Field soil classification, visual examination, field oil sheen test, odor observations, field headspace organic vapor screening	One sample to characterize each different-appearing soil encountered; periodic sampling for assessing continuity of similar-appearing soils.
	Characterize nature of key chemical constituents in identified source area wastes/soils	12 of 25	PAHs, VOCs	One sample to characterize each distinct type of visual contamination; assess different levels of visual contamination.
	As above, plus assess levels of phenols and possible correlation with PAHs	5 of 25	PAHs, VOCs, Phenols	Approximately 20% of samples analyzed for PAHs; assess different levels of visual contamination.
	Characterize nature of key chemical constituents associated with thionizer process	3 of 25	PAHs, VOCs, Arsenic, Cyanide	From trenches near Thionizer Building.
	Assess full-range of chemical constituents in identified source area waste/soil	4 of 25	Full-Scan ⁽¹⁾	One from apparent coal-tar waste; one from apparent contamination in new slip area; one from each shallow boring in former pond areas.
	Assess full-range of chemical constituents and leaching characteristics of compacted coal fines layer	1 of 25	Full-Scan ⁽¹⁾ , TCLP	From compacted coal fines layer in slip area.
Background Soil Sampling	Assess off-site concentrations of full-range of chemical constituents in soils at surrounding industrial and non-industrial locations	8	Full-Scan ⁽¹⁾	Pre-Determined Locations
Surficial Soil Sampling	Assess nature of on-site soils relative to full-range of chemical constituents	17	Full-Scan ⁽¹⁾	Pre-Determined Locations
Groundwater Sampling	Characterize groundwater quality	10	Full-Scan ⁽¹⁾	New and Pre-Existing Monitoring Wells

⁽¹⁾Semivolatiles, VOCs, Metals, PCBs, Pesticides

TABLE 3.5-1
GENERAL RI/FS OBJECTIVES

Objective	RI Activity	FS Activity
Characterize extent of MGP and creosote contamination.	Investigate horizontal and vertical extent of key contaminants in suspected source areas and suspected transport pathways.	Evaluate applicability of no-action alternative for source areas/pathways.
Characterize non-MGP and non-creosote types of contaminants.	Investigate "nature" of contaminants in source areas and in pathways; identify contaminants not anticipated at MGP and creosoting facilities.	Evaluate environmental/public health threat, identify remedial technologies, distinguish between likely sources of the identified contaminants.
Characterize magnitude of soil contamination.	Estimate key contaminant concentration isopleths, interpret the collected data.	Evaluate costs to achieve applicable or relevant and appropriate standards.
Characterize magnitude of water contamination.	Estimate key contaminant concentration isopleths, interpret the collected data.	Evaluate costs to achieve applicable or relevant and appropriate standards.
Characterize contaminant release and transport mechanism.	Investigate likely pathways/transport routes, transport medium, contaminant transport properties, identify potential receptors.	Identify most effective points in pathway to control transport of contaminants.
Assess environmental/public health factors.	Investigate likely routes of exposure, identify environmental and public health threat.	Evaluate applicable standards of risk, identify constraints on applicable remediation technologies.

TABLE 3.4-5
NON-CLP ANALYTICAL PARAMETERS AND
QUANTITATION LIMITS

<u>Non-CLP Parameters</u>	<u>Quantitation Limit</u>	
	<u>Water ($\mu\text{g/L}$)</u>	<u>Soil ($\mu\text{g/kg}$)</u>
Benzene	1.0	1.0
Toluene	1.0	1.0
Ethyl Benzene	1.0	1.0
Xylene	1.0	1.0
Biochemical Oxygen Demand	10,000	NA
Chemical Oxygen Demand	20,000	NA
Oil and Grease	1,000	NA
Total Suspended Solids	4,000	NA
Low Level PAHs:		
Naphthalene	0.005	NA
2-Methylnaphthalene	0.005	NA
Acenaphthylene	0.005	NA
Acenaphthene	0.005	NA
Dibenzofuran	0.005	NA
Fluorene	0.005	NA
Phenanthrene	0.005	NA
Anthracene	0.005	NA
Fluoranthene	0.005	NA
Pyrene	0.005	NA
Benzo(a)anthracene	0.005	NA
Chrysene	0.005	NA
Benzo(b&k)fluoranthene	0.005	NA
Benzo(a)pyrene	0.005	NA
Indeno(1,2,3-cd)pyrene	0.005	NA
Dibenzo(a,h)anthracene	0.005	NA
Benzo(g,h,i)perylene	0.005	NA

TABLE 3.4-4 (continued)
CLP ANALYTICAL PARAMETERS AND QUANTITATION LIMITS

INORGANIC TARGET ANALYTE LIST (TAL)

Analyte	Contract Required Detection Limit (1.2) (ug/L)
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	3
Magnesium	5000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5000
Selenium	5
Silver	10
Sodium	5000
Thallium	10
Vanadium	50
Zinc	20
Cyanide	10

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stopper the flask, and gently turn the flask
end-over-end three times. Transfer the
standard to a 1-ml conical vialx#^P^N^V0*%%
cKed

40°C (3 min) -----> 8°C/min
200°C (25 min)

Capillary

10°C (8 min) -----> 4°C/min
150°C (5 min)

NOTE: A fast ramp of the capillary column to 220°C
at data acquisition can be useful for boiling off
late eluting non-target analytes.

5.1.2 Cryofocusing Unit Parameters

Focus Temperature - 150°C
Injection - 150 to 150 in 0.75 min.

5.1.3 Injector temperature - 220°C

5.1.4 Detector temperature - 250°C

5.1.5 Carrier gas flow - 25 mL/min (Packed)
- 2 mL/min (Capillary)

5.2 Purge and Trap

5.2.1 Purge flow - 40 mL/min

5.2.2 Purge time - 11 minutes

5.2.3 Desorb time - 4 minutes

5.2.4 Desorb temperature - 180°C

5.2.5 Bake time - 10 minutes minimum

5.2.6 Bake temperature - 220°C

5.3 Photoionization Detector

5.3.1 Input attenuation - 100

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5.3.2 Coarse offset - 60

5.3.3 Heater temperature - approx. 200°C Do not exceed 250°C.

5.4 Hall Detector

5.4.1 Reaction temperature - 900°C

5.4.2 H₂ gas flow - approx. 50 mL/min

5.4.3 Detector range - 100

6.0 Initial Calibration Procedures

6.1 The linear calibration range of the instrument must be determined before the analysis of any samples. The instrument must be calibrated using the same instrument conditions that are used to analyze the samples. The following special conditions must be considered before performing initial calibration:

- If 25 mLs of water is purged to achieve lower detection limits, then both the initial and continuing calibrations must be performed by purging 25 mL of the aqueous standards.
- If soils/sediments are purged using the heated purge technique, then both the initial and continuing calibration must be performed using heated purge.
- If soils/sediments are analyzed using the high-level method(methanol) extraction, the calibration curve and all continuing calibration checks must incorporate a total of 100 ul of methanol into each calibration point.

6.2 Prepare aqueous calibration standards at a minimum of five concentration levels for each parameter analyzed. The concentrations of the aqueous standards should bracket the linear range of the instrument. For most compounds, the calibration standards are at 5 ppb, 20 ppb, 40 ppb, 100 ppb, and 200 ppb. For compounds that co-elute, such as M and P-Xylene, each of the two compounds is at the indicated concentration. Tert butyl methyl ether(TBME) levels are 10 ppb, 40 ppb, 80 ppb, 200 ppb, and 400 ppb.

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- 4.3.3 Targets 3. Accustandard M-601C, containing 2-chloroethyl vinyl ether, at 200 ug/ml. Solution should be replaced one week after opening, or before vendor supplied expiration date, whichever comes first. Four vials x 1 ml.
- 4.3.4 Targets 4. Accustandard M-501 Trihalomethanes mixture, containing chloroform, dichlorobromomethane, bromoform, and dibromochloromethane, at 200 ug/ml. Solution should be replaced one month after opening, or before vendor supplied expiration date, whichever comes first. One vial x 1 ml.
- 4.3.5 Targets 5. Accustandard M-602-GAS, containing Method 602/8020 target analytes at 200 ug/ml. This solution will be used only when conducting analysis of Method 602/8020 target analytes alone. Solution should be replaced one month after opening, or before vendor supplied expiration date, whichever comes first. One vial x 1 ml.
- 4.3.6 Surrogates 1. Accustandard M-001R-1, 601 surrogate, containing bromochloromethane at 4000 ug/ml. Solution should be replaced one month after opening, or before vendor supplied expiration date, whichever comes first. One vial x 1 ml.
- 4.3.7 Surrogates 2. Accustandard M-602-SS, 602 surrogate, containing a,a,a-trifluorotoluene, at 200 ug/ml. Solution should be replaced one month after opening, or before vendor supplied expiration date, whichever comes first. One vial x 1 ml.
- 4.3.8 Internal Standard. Accustandard CLP-004-100X, containing 4-bromofluorobenzene at 2500 ug/ml. Solution should be replaced one month after opening, or before vendor supplied expiration date, whichever comes first. One vial x 1 ml.

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4.4 Working Standard Solutions

Using the stock solutions, prepare the following working standard solutions by making appropriate dilutions in methanol.

- 4.4.1 Calibration standards - Use a 2-ml ClassA volumetric flask rinsed three times with methanol. Add 500 ul or less of methanol to the flask, and add 250 ul of each of solutions Targets 1 to 4, or 250 ul of Targets 5 (if calibration is for Methods 602/8020). Add methanol to the mark, stopper the flask, and gently turn the flask end-over-end three times. Transfer the standard to a 2-ml conical vial and cap with a Teflon-lined stop-go cap. The final concentration of this solution is 25 ug/ml.

Calibration solutions should be prepared fresh weekly, keeping in mind that the holding times for Target solutions 1 to 4 range from one week to one month. The calibration standards working solution may also be used for matrix spiking purposes.

- 4.4.2 Surrogate Spike Standards 1 - Use a 2-ml Class A volumetric flask rinsed three times with methanol. Add 1 ml or less of methanol to the flask, add 12.5 ul of Surrogates 1, and 250 ul of Surrogates 2. Add methanol to the mark, stopper the flask, and gently turn the flask end-over-end three times. Transfer the standard to a 2-ml conical vial and cap with a Teflon-lined stop-go cap. The final concentration of this solution is 25 ug/ml.

Surrogate spike standards should be prepared fresh monthly. This solution is for use with water and low level soil/sediment samples.

- 4.4.3 Surrogate Spike Standards 2 - Use a 1-ml Class A volumetric flask rinsed three-times with methanol. Add 250 ul or less of methanol to the flask, add 25 ul of Surrogates 1, and 500 ul of Surrogates 2. Add methanol to the mark,

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3.2 Purge and trap system

- 3.2.1 Ten-Station automated purge device Tekmar Model ALS or equivalent for water sample analyses.
- 3.2.2 Ten-Station automated heated purge device Tekmar Model 4200 or equivalent.
- 3.2.3 Purge and trap device - Tekmar LSC-2 or equivalent.

3.3 Gas Chromatograph - Varian Model 3740 or equivalent.

3.4 Data System - Varian Model 402 or DS654 or equivalent.

3.5 Detectors

- 3.5.1 HNU Model PI52 with a 10.2 eV lamp or equivalent.
- 3.5.2 Tracor Model 700A Hall Detector or equivalent.

3.6 Gas Chromatographic Columns

Column 1 - 30 M x 0.534 mm ID DB-1, 5 uM film coating.

Column 2 - 30 M x 0.534 mm ID DB-624, 3 uM film thickness, J & W Scientific or equivalent.

3.7 Trap - Commercially prepared per specifications in Method 5030. The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch. The trap must be packed with 1/3 each of Tenax, silica gel, and 1/3 charcoal.

3.8 Syringes - Hamilton gas tight syringes

- 3.8.1 uL Syringes - 10, 25, 50, 100, 500 and 1000 uL.
- 3.8.2 mL Syringes - 5, 10 and 25 mL.

3.9 Volumetric Flasks - 10, 25, 50, 100 and 1000 mL.

3.10 A heater or heated water bath capable of maintaining the sparging vessel at 40°C.

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3.11 Cryofocusing Unit - Tekmar Capillary Interface or equivalent.

4.0 Reagents

4.1 Methanol - Purge and Trap Methanol obtained for Burdick and Jackson or equivalent. The methanol must be demonstrated to be free of volatile target analytes.

4.2 Reagent Water - Organic free water in which an interferant is not observed at the method detection limits, prepared by passing tap or deionized water through a carbon filter, followed by bubbling with a contaminant-free inert gas for one hour.

4.3 Stock Standard Solutions

Stock standard solutions can be purchased in a kit from Accustandard (25 Science Park, suite 687, New Haven, Ct. 06511, 203-786-5290) containing the solutions listed below. Each solution will have a vendor supplied expiration date and a certificate of analysis. The solutions described below are applicable only for calibration and matrix spiking purposes. Independently prepared LCS standards should be obtained from a separated source.

4.3.1 Targets 1. Accustandard M-601A/M-602-GAS mix containing target compounds for Methods 601, 602, 8010A, 8020A, and for BTEX analyses with the exception of gases, 2-Chloroethyl vinyl ether, and Trihalomethanes, at 200 ug/ml. This mix is specially prepared for CH2M HILL. Solution should be replaced one month after opening, or before vendor supplied expiration date, whichever comes first. One vial x 1 ml.

4.3.2 Targets 2. Accustandard M-601B Gases mixture, containing bromomethane, chloromethane, chloroethane, dichlorodifluoromethane, trichlorofluoromethane, and vinyl chloride, at 200 ug/ml. Solution should be replaced one week after opening or before vendor supplied expiration date, whichever comes first. Four vials x 1 ml.

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STANDARD OPERATING PROCEDURE

EPA METHODS 8010, 8020

1.0 Summary of the Methods

This standard operating procedure describes the procedures used to analyze water, soil and sediment samples by using purge and trap gas chromatographic techniques for extraction and analysis of selected volatile compounds.

Water samples are analyzed directly by bubbling an inert gas through a 5 mL or 25 mL sample contained in a specially designed purging chamber at ambient temperature.

Soil and sediment samples are extracted and analyzed by one of two methods depending on the concentration of volatile components in the matrix. For low level solid samples, the matrix is suspended in water, and the suspension is purged in a heated sparging chamber. High level solid samples are extracted with methanol. An aliquot of the methanol extract is spiked into reagent water and analyzed according to procedures described for water samples.

The volatile components from the different matrices are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatiles are trapped. After purging is completed, the sorbent column is heated and backflushed with an inert gas. The volatile components are transferred onto a gas chromatographic column which is temperature programmed to separate the compounds. The gas chromatograph is equipped with a photoionization detector connected in series with a Hall detector. The photoionization detector detects the aromatic compounds, and the Hall detector detects the halogenated compounds.

- 1.1 Working Linear Range - The working linear range for the method is 1 ppb - 200 ppb for most compounds.
(see the copies of the 5-point calibration forms in attachment A).

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1.2 Instrument Detection Limits - Instrument Detection Limit Studies are run after a new column is installed or after any major change in the GC system. Copies of the most recent IDL studies for GC #1 (DB-624 megabore column; file 1DL0220A) and GC#2 (DB-1 megabore column; file 2DL0217A) are found in attachment B.

2.0 Target analytes and Method Detection Limits

Tables 1 and 2 list the target analytes and the nominal estimated detection limits for the analysis of water samples. The detection limits must be verified by performance of validation experiments. The validation process requires the analysis of a minimum of seven replicates of a known concentration of the compounds in water. The concentration of the replicate analyses must be one to five times the estimated nominal detection limits. The estimated detection limit is calculated by multiplying the standard deviation of the replicate results (in ug/L) by three. The reported nominal detection limits are valid if they are larger than the three standards deviation values. This verification procedure must be performed separately for analysis of 5 mL and 25 mL water samples.

The soil sample detection limits are adjusted to account for the differences in the mass of the sample extracted and the dilution of the extract for medium and high level analyses.

Method performance at concentrations well within the calibration range of the method must be determined to provide recovery ranges for quality assurance objectives. According to directions given in 40 CFR Part 136, at least 4 spikes must be performed to determine accuracy ranges for the different analytes. Using this procedure, the accuracy ranges are calculated from the mean and plus or minus 2 standard deviations.

3.0 Apparatus and Materials

3.1 Sample Containers - 40 mL vials with a screw cap with a hole in the center of the cap. A Teflon-faced silicone septum is placed inside the screw cap so only the Teflon comes into contact with the water samples. The vials and septa are routinely purchased from I-Chem and are not reused.

Standard Operating Procedure

EPA Methods 8010/8020

October 1991

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Written By: Mary Wisdom

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Table 1: Method 8010 Halocarbons and MDL

Table 2: Method 8020 Halocarbons and MDL

Table 3: EPA 601 Matrix Spike Acceptance Criteria

Table 4: EPA 602 Matrix Spike Acceptance Criteria

Attachment A: Initial and Continuing Calibration Data

Attachment B: Instrument Detection Limit Data

TABLE II
(Cont'd)

Procedure	Container	Quantity	Preservation	Holding Time
VOLATILE ORGANICS				
601/8010 Purgeable Halocarbons	G-TLS	3@40 ml	Cool @ 4°C; HCl ^a	14 days
602/8020 Purgeable Aromatics	G-TLS	3@40 ml	Cool @ 4°C; HCl ^a	14 days
601/602; 8010/8020	G-TLS	3@40 ml	Cool @ 4°C; HCl ^a	14 days
603/8015 Nonhalogenated Volatile Organics	G-TLS	3@40 ml	Cool @ 4°C; HCl ^a	14 days
624/8240 Volatile Organics	G-TLS	3@40 ml	Cool @ 4°C; HCl ^a	14 days
BTXE	G-TLS	3@40 ml	Cool @ 4°C; HCl ^a	14 days
TFH as gas	G-TLS	3@40 ml	Cool @ 4°C; HCl ^a	14 days
TFH as gas + BTXE	G-TLS	3@40 ml	Cool @ 4°C; HCl ^a	14 days
Trihalomethane	G-TLS	2@40 ml	Cool @ 4°C; Na ₂ S ₂ O ₃	14 days
SEMI-VOLATILE ORGANICS				
604/8040 Phenols	G-TLC	1@2.5 liters	Cool @ 4°C	7 days ^b
608/8080 Pesticides and PCBs	G-TLC	1@2.5 liters	Cool @ 4°C	7 days ^b
610/8010 PAH/PNA	G-TLC	1@2.5 liters	Cool @ 4°C	7 days ^b
612/8120 Chlorinated Hydrocarbons	G-TLC	1@2.5 liters	Cool @ 4°C	7 days ^b
622/8140 Organophosphorus Pesticides	G-TLC	1@2.5 liters	Cool @ 4°C	7 days ^b
615/8150 Chlorinated Herbicides	G-TLC	1@2.5 liters	Cool @ 4°C	7 days ^b
613/8280 2,3,7,8-TCDD	G-TLC	2@2.5 liters	Cool @ 4°C	7 days ^b
625/8250 Semivolatile Organics	G-TLC	2@2.5 liters	Cool @ 4°C	7 days ^b
^a Added to 2 vials only - pH<2; ^b days to extraction; 40 days to analysis after extraction P - Polyethylene; G - Glass; TLC - Teflon lined cap; TLS - Teflon lined septum				

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TABLE II
(Cont'd)

NTS				
Procedure	Container	Quantity	Preservation	Holding Time
SEMI-VOLATILE ORGANICS (CONTINUED)				
504 EDB/DBCP	G-TLS	2@125 ml	Cool @ 4°C	28 days ^b
TFH: diesel, jet fuel, kerosene	G-TLC	1@2.5 liters	Cool @ 4°C	7 days ^b
METALS				
Chromium VI	P,G	250 ml	Cool @ 4°C	24 hrs
Mercury	P,G	250 ml	Cool @ 4°C; HNO ₃ ^a	28 days
Organic Lead	G-TLC	1 liter	Cool @ 4°C	6 months
All other metals	P,G	1 liter	Cool @ 4°C; HNO ₃ ^a	6 months
^a pH<2; ^b days to extraction; 40 days to analysis after extraction P - Polyethylene; G - Glass; TLC - Teflon lined cap; TLS - Teflon lined septum				

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TABLE II

Procedure	Container	Quantity	Preservation	Holding Time
GENERAL AND INORGANICS				
Acidity	P,G	250 ml	Cool @ 4°C	14 days
Alkalinity	P,G	250 ml	Cool @ 4°C	14 days
Ammonia	P,G	1 liter	Cool @ 4°C; H ₂ SO ₄ ^c	28 days
BOD	P,G	1 liter	Cool @ 4°C	48 hours
Boron	P,G	100 ml	Cool @ 4°C	28 days
Bromide	P,G	250 ml	Cool @ 4°C	28 days
COD	P,G	100 ml	Cool @ 4°C; H ₂ SO ₄ ^c	28 days
Chlorine, residual	P,G	250 ml	Cool @ 4°C	24 hours
Chloride	P,G	250 ml	Cool @ 4°C	28 days
Color	P,G	250 ml	Cool @ 4°C	48 hours
Coliform	P,G	125 ml	Cool @ 4°C; Na ₂ S ₂ O ₃	6 hours
Cyanide	P,G	1 liter ^a	Cool @ 4°C; NaOH ^b	14 days
Flashpoint	G-TLC	250 ml	Cool @ 4°C	28 days
Fluoride (total)	P	250 ml	Cool @ 4°C	28 days
Hardness	P,G	250 ml	Cool @ 4°C; H ₂ SO ₄ ^c	6 months
Nitrate	P,G	100 ml	Cool @ 4°C	48 hours
Nitrite	P,G	100 ml	Cool @ 4°C	48 hours
Nitrate/Nitrite	P,G	100 ml	Cool @ 4°C; H ₂ SO ₄ ^c	28 days
Oil and Grease	G-TLC	1 liter ^a	Cool @ 4°C; H ₂ SO ₄ ^c	28 days
Phenolics (4AAP)	G	500 ml	Cool @ 4°C; H ₂ SO ₄ ^c	28 days
Phosphorus, total	P,G	250 ml	Cool @ 4°C; H ₂ SO ₄ ^c	28 days
Phosphate, ortho	P,G	250 ml	Filter immediately; Cool @ 4°C	48 hours
^a Need 3 liters for matrix spike and matrix spike duplicate; ^b pH>12; ^c pH<2 P - Polyethylene; G - Glass; TLC - Teflon lined cap; TLS - Teflon lined septum				

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TABLE II
(Cont'd)

Procedure	Container	Quantity	Preservation	Holding Time
GENERAL AND INORGANICS (CONTINUED)				
pH	P,G	100 ml	Cool @ 4°C	Immediately
Radiological	P,G	1 liter	Cool @ 4°C; HNO ₃	6 months
Reactivity in acids and bases	P,G	500 ml	Cool @ 4°C	7 days
Reactivity in water	P,G	250 ml	Cool @ 4°C	28 days
Silica	P	250 ml	Cool @ 4°C	28 days
Solids, dissolved	P,G	250 ml	Cool @ 4°C	7 days
Solids, suspended total and volatile	P,G	1 liter	Cool @ 4°C	7 days
Solids, total and volatile	P,G	250 ml	Cool @ 4°C	7 days
Specific Conductivity	P,G	500 ml	Cool @ 4°C	28 days
Specific Gravity	P,G	25 ml	Ambient	28 days
Steel Corrosion	P,G	500 ml	Cool @ 4°C	28 days
Sulfate	P,G	500 ml	Cool @ 4°C	28 days
Sulfite	P,G	500 ml	Cool @ 4°C	24 hours
Sulfide	P,G	500 ml	Cool @ 4°C; Zn(Ac); NaOH	7 days
Surfactants, MBAS	P,G	1 liter	Cool @ 4°C	48 hours
Tannins and Lignin	G	1 liter	Cool @ 4°C; NaOH - pH>9	7 days
TKN	P,G	1 liter	Cool @ 4°C; H ₂ SO ₄ ^a	28 days
Total Organic Carbon	P,G	250 ml	Cool @ 4°C; H ₂ SO ₄ ^a	28 days
Total Organic Halogen	G-TLC	500 ml	Cool @ 4°C; H ₂ SO ₄ ^a	28 days
Total Petroleum Hydrocarbons	G-TLC	1 liter ^b	Cool @ 4°C; H ₂ SO ₄ ^a	28 days
Turbidity	P,G	500 ml	Cool @ 4°C	48 hours
^a pH<2; ^b Need 3 liters for matrix spike and matrix spike duplicate P - Polyethylene; G - Glass; TLC - Teflon lined cap; TLS - Teflon lined septum				

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SAMPLE KIT SOP

Revision 1
June 11, 1991

Date: _____

By: _____

**Table 1 (Cont'd)
CUSTODY INVENTORY
PLASTIC**

Vender/Stock #	#/Package	Size	Description	# Cases In Stock	Cases of Lids In Stock	Reorder Point	# Ordered
Smith 20236/72081	Package 495	250 ml.	Plastic Bottle			2 cases	
Smith 20258/72083	Package 283	500 ml.	Plastic Bottle			3 cases	
Smith 20274/72087	Package 140	1,000 ml.	Plastic Bottle			4 cases	
Plastics Inc.	Package 48	½ Gal.	Plastic Jug			1 cases	
Plastics Inc.	Package 25	1 Gal.	Plastic Jug			1 cases	
VWR16129-028	Package 12	250 ml	Fecal Bottles			1 cases	
IChem 319-1000 EP 150-01W/WM	Package 12	1,000 ml	Cation Bottles (Q.C.)			20 cases	
IChem 319-500 EP 150-500W/WM	Package 24	500 ml	Cation Bottles (Q.C.)			12 cases	

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Table 1 (Cont'd)
MISCELLANEOUS CUSTODY SUPPLIES

Vendor/Stock #	#/Package	Size	Description	# in Stock	Reorder Point	# Ordered
Acon	Bundle 25	11x10x10	Cardboard Box		1 cs	
	Bundle 20	15x12x10	Cardboard Box		1 cs	
	Bundle 20	18x14x14	Cardboard Box		1 cs	
American Packaging	750 ft roll	12"x12" perforated	Bubble Pack		1 roll	
		20 cu ft ³ /bag	Foam Peanuts		1 bag	
CH2M HILL GNV/Reprographics	Order by the thousand	Small	Custody Seals and Labels		1,000	
	Order by the thousand	Large	Custody Seals and Labels		1,000	
Davis Printing	500 3-part forms		Sample Kit Request Forms		500	
			Labels (backup supplier)			
National Bag 87-825A2	Case of 36 rolls	2" x 55 yds	Packing Tape		18 rolls	
National Bag 87-644A2	Order 6 rolls	4" x 72 yds	Label Tape		1 roll	
National Bag 87-658A2	Case of 36 rolls	1" x 180 yds	Strapping Tape		18 rolls	
Plasti-Kraft UPC # 00111	24 per pkg	11 qt	Cooler		1 cs	
Plasti-Kraft UPC # 00622	12 per pkg	22 qt	Cooler		1 cs	
Plasti-Kraft UPC # 00228	12 per pkg	28 qt	Cooler		2 cs	
Sam's Wholesale Club			Shipping Coolers			
VWR 32915-770	Package of 50	Small	Vinyl Gloves		1 box	
VWR 32915-772	Package of 50	Large	Vinyl gloves		1 box	
VWR 14673-010	Case of 1000		Disposable Pipets		1 box	
VWR 53281-724	Case of 500		Disposable Pipets		1 box	
VWR 60786-028		10 ml	pH Paper		1 roll	

FORM C
CUSTODY SEAL



CUSTODY SEAL

Date

Signature

SAMPLE KIT SOP

Revision 1

June 11, 1991

Date: _____

By: _____

Table 1
CUSTODY INVENTORY
GLASS

Vender/Stock #	#/Package	Size	Description	# Cases In Stock	Cases of Lids In Stock	Reorder Point	# Ordered
IChem 320-006 EP 130-02C	Package 24	2 oz.	Glass Jar Series			12 cases	
IChem 220-0060	Package 24	2 oz.	w/Teflon Lid Series 200			12 cases	
VWRIR320-0250 E-P 131-08C	Package 12	8 oz.	Glass Jar w/Teflon Lid			12 cases	
Smith 10159/72097	Package 12	16 oz.	Glass Jar (short)			12 cases	
Smith 10174/72097	Package 12	32 oz.	Glass Jar (tall)			12 cases	
IChem S2349-0125 E-P 5114-125A	Package 12	Amber/Flint (Teflon Septum) 4 oz. For EDB (125) (Regular)				5 cases	
Smith 10960/72155	Package 12	16 oz.	Amber-500 ml TOX and Phenol			10 cases	
Smith 10976/72159	Package 12	32 oz.	1000 ml Amber			6 cases	
VWR 2123/16161-279	Package 12	32 oz.	1,000 ml. Amber (teflon)			12 cases	
EP 110-80A VWR IR345-2360	Package 6	80 oz.	2,000 ml. Amber			40 cases	
83-9740-100cr	Package 100	40 ml.	VOA vials w/Septa			5 cases	
IChem 320-1000 (QC) EP 133-32C (QC)	Package 12	32 oz	Glass Jar (Tall)			6 cases	
Sci. Prd. B7503-7	Package 72		Teflon Lids for TOX			1 case	



FORM A
QUALITY ANALYTICAL LABORATORIES
SAMPLE KIT REQUEST

SAMPLE KIT SOP

Revision 1

June 11, 1991

DATE REQUESTED : _____

NEEDED BY : _____

FIELD EVENT DATES: _____

PROJECT CONTACT : _____

SHIPPING CHARGES: _____

FOR LAB USE ONLY

PROJECT NAME _____

PROJECT # _____

QUOTATION # _____

TURN AROUND TIME _____

MULTIPLIER _____

QC LEVEL 1 2 3 _____

SDWA NPDES RCRA HAZWRAP NEESA

☐ ☐ ☐ ☐ ☐

NO. OF SAMPLES: _____ MATRIX: _____ SAMPLING SITES: _____

ANALYSES: _____

NO. OF CONTAINERS	TYPES OF CONTAINERS	MATRIX	ANALYSES REQUESTED
_____	40 ml vial w/teflon septum	Water	_____
_____	40 ml vial w/teflon septum w/ _____	Water	_____
_____	125 ml amber bottle w/teflon septum (pair)	Water	_____
_____	500 ml amber bottle w/ _____	Water	_____
_____	1 L amber bottle w/teflon w/ _____	Water	_____
_____	2 L amber bottle w/teflon	Water	_____
_____	2 L amber bottle w/teflon	Water	_____
_____	Other: _____	_____	_____
_____	250 ml plastic bottle w/H ₂ SO ₄	Water	_____
_____	500 ml plastic bottle w/H ₂ SO ₄	Water	_____
_____	1 L plastic bottle w/H ₂ SO ₄	Water	_____
_____	500 ml plastic bottle w/O	Water	_____
_____	1 L plastic bottle w/O	Water	_____
_____	500 ml plastic bottle w/NaOH + ZnAc	Water	_____
_____	500 ml plastic bottle w/HNO ₃	Water	_____
_____	1 L plastic bottle w/HNO ₃	Water	_____
_____	½ gal plastic jug w/ _____	Water	_____
_____	Other: _____	_____	_____
_____	2 oz glass jar w/teflon	Soil	_____
_____	4 oz glass jar w/teflon	Soil	_____
_____	8 oz glass jar w/teflon	Soil	_____
_____	8 oz glass jar w/teflon	Soil	_____
_____	16 oz glass jar	Soil	_____
_____	32 oz glass jar	Soil	_____
_____	Other: _____	_____	_____

SHIP TO: _____ COC FORM: _____ SHIPPING LABELS: _____

ADDRESS: _____ CUSTODY SEALS: _____

CITY, STATE: _____ ZIP: _____ DATE SHIPPED: _____

ATTENTION: _____ PHONE: _____ SHIPPED BY: _____

CHM HILL QUALITY ANALYTICS
CHAIN OF CUSTODY RECORD

SAMPLE KIT SOP
Revision 1
June 11, 1991

PROJECT NUMBER			PROJECT NAME			# OF CONTAINERS	CLIENT ADDRESS AND PHONE NUMBER										LAB ID	FOR LAB USE ONLY				
CLIENT NAME							ANALYSES REQUESTED											LAB#				
PROJECT MANAGER				COPY TO:														LAB#				
REQUESTED COMP. DATE				SAMPLING REQUIREMENTS														PROJECT NO.				
				SDWA <input type="checkbox"/> NPDES <input type="checkbox"/> RCRA <input type="checkbox"/> OTHER <input type="checkbox"/>														ACK		VERIFIED		
STA NO.	DATE	TIME	COMP	GRA	SOIL	SAMPLE DESCRIPTIONS (12 CHARACTERS)											QUOTE#		BS			
																	NO. OF SAMP		PG	OF		
																	REMARKS					
SAMPLED BY AND TITLE			DATE/TIME			RELINQUISHED BY					DATE/TIME					HAZWRAP/NEESA Y N						
RECEIVED BY:			DATE/TIME			RELINQUISHED BY:					DATE/TIME					QC LEVEL 1 2 3						
RECEIVED BY:			DATE/TIME			RELINQUISHED BY:					DATE/TIME					COC		ICE				
RECEIVED BY LAB:			DATE/TIME			SAMPLE SHIPPED VIA					AIR BILL#					ANA REQ		TEMP				
						UPS BUS FED-EX HAND OTHER										CUST SEAL		Ph				
																SAMPLE COND.						
REMARKS													ENTERED INTO LIMS					COC REVIEWED				

FORM B

STANDARD OPERATING PROCEDURE

1.0 SAMPLE KIT PREPARATION

1.1 SAMPLE CONTAINERS

Sample containers and other items are prepared as a sample kit according to the sample kit request form (see Form A).

Sample containers and other supplies are purchased from approved vendors and stored in the warehouse or sample custody area of the laboratory. Inventories are maintained so that kits can be filled with short notice (see Inventory Table I).

Containers for projects involving higher level QC such as CLP, HAZWRAP, or NEESA, are purchased precleaned.

1.2 PRECLEANED SAMPLE CONTAINERS

Recommended CLP TAL (metals and cyanide) and CLPTCL (VOA, Semi-VOA, Pest/PCB) containers are supplied with certificates of analysis. Notations documenting container use are made directly on certificates. These certificates are retained on file in sample custody by lot number and type.

Other container types are assigned coded lot numbers as soon as new shipments arrive. When containers are needed for higher QC level projects, the appropriate kind and number of containers are reserved for specific parameters, and 1 percent of these containers are submitted for analysis. These reports are retained on file in Sample Custody by project and date.

1.3 SAMPLE KIT CONTENTS

Labeled sample bottles, jars, and other supplies are provided in shipping containers. Usually hard-shell ice chests or coolers are used so that samples may be shipped at 4°C, if necessary. A typical sample kit includes the following materials.

Sample bottles or jars

A list of most containers for water analysis is posted in the sample custody area and is available to personnel who are packing kits or filling out kit requests, as needed (See Table II).

- a. Water samples often require pH adjustment for preservation. Preservative solutions are prepared by trained personnel and are provided in appropriately labeled sample bottles.
- b. Soil containers are not so varied and do not require preservation by pH adjustment. Wide mouth jars in several sizes are available.

Paperwork

Paperwork is shipped in a resealable plastic bag inside the cooler

- a. A copy of the sample kit request form is included for the client's use (see Form A).
- b. A chain-of-custody form is provided and also custody seals, if required by the QC level of the project or by the client's request (See Form B, C.)
- c. Additional instructions, memos, or quotations also may be included.

1.4 KIT SHIPMENT

Kit shipment is by the most economical means that will meet the client's sampling schedule. Under certain rush circumstances, the client may be required to pay the shipping costs.

The person responsible for preparing and sending the sample kit should sign and date the sample kit request form in the lower right corner.

The kit request form should be filed with the other completed forms on the monthly clipboard kept in Sample Custody.

Appendix B

Laboratory Procedures

3. Do they remain legible even if wet? (Y/N) _____

B. Sample Seals:

1. Are sample seals placed on those containers to ensure the samples are not altered? (Y/N) _____

C. Field Logbook:

1. Is a field logbook maintained? (Y/N) _____

2. Does it document the following:

a. Purpose of sampling (e.g., detection or assessment? (Y/N) _____

b. Identification of well? (Y/N) _____

c. Total depth of each well? (Y/N) _____

d. Static water level depth and measurement technique? (Y/N) _____

e. Presence of immiscible layers and detection method? (Y/N) _____

f. Collection method for immiscible layers and sample identification numbers? (Y/N) _____

g. Well yield - high or low? (Y/N) _____

h. Purge volume and pumping rate? (Y/N) _____

i. Time well purged? (Y/N) _____

j. Well evacuation procedures? (Y/N) _____

k. Sample withdrawal procedure? (Y/N) _____

l. Date and time of collection? (Y/N) _____

m. Well sampling sequence? (Y/N) _____

n. Types of sample containers and sample identification numbers? (Y/N) _____

o. Preservative(s) used? (Y/N) _____

p. Parameters requested? (Y/N) _____

q. Field analysis data and method(s)? (Y/N) _____

r. Sample distribution and transporter? (Y/N) _____

s. Field observations:

■ Unusual well recharge rates? (Y/N) _____

■ Equipment malfunction(s)? (Y/N) _____

■ Possible sample contamination? (Y/N) _____

■ Sampling rate? (Y/N) _____

t. Field team members? (Y/N) _____

u. Climatic conditions and air temperature? (Y/N) _____

D. Chain-of-Custody Record:

1. Is a chain-of-custody record included with each sample? (Y/N) _____

2. Does it document the following:

a. Sample number? (Y/N) _____

b. Signature of collector? (Y/N) _____

c. Date and time of collection? (Y/N) _____

2. Are samples for the following analyses field acidified to pH<2 with HNO₃:
 - a. Iron? (Y/N) _____
 - b. Manganese? (Y/N) _____
 - c. Sodium? (Y/N) _____
 - d. Total metals? (Y/N) _____
 - e. Dissolved metals? (Y/N) _____
 - f. Radium? (Y/N) _____
 - g. Gross alpha? (Y/N) _____
 - h. Gross beta? (Y/N) _____
 3. Are samples for the following analyses field acidified to pH<2 with H₂SO₄:
 - a. Phenols? (Y/N) _____
 - b. Oil and grease? (Y/N) _____
 4. Is the sample for TOC analyses field acidified to pH<2 with H₂SO₄ or HCl? (Y/N) _____
 5. Is the sample for TOX analysis preserved with 1 ml of 1.1 M sodium sulfite? (Y/N) _____
 6. Is the sample for cyanide analysis preserved with NaOH to pH>12? (Y/N) _____
- C. Special Handling Considerations:
1. Are organic samples handled without filtering? (Y/N) _____
 2. Are samples for volatile organics transferred to the appropriate vials to eliminate headspace over the sample? (Y/N) _____
 3. Are samples for metal analysis split into two portions? (Y/N) _____
 4. Is the sample for dissolved metals filtered through a 0.45 micron filter? (Y/N) _____
 5. Is the second portion not filtered and analyzed for total metals? (Y/N) _____
 6. Is one equipment blank prepared each day of groundwater sampling? (Y/N) _____

III. REVIEW OF CHAIN-OF-CUSTODY PROCEDURES

- A. Sample Labels:
1. Are sample labels used? (Y/N) _____
 2. Do they provide the following information:
 - a. Sample identification number? (Y/N) _____
 - b. Name of collector? (Y/N) _____
 - c. Date and time of collection? (Y/N) _____
 - d. Place of collection? (Y/N) _____
 - e. Parameter(s) requested? (Y/N) _____

II. REVIEW OF SAMPLE PRESERVATION AND HANDLING PROCEDURES

A. Sample Containers:

1. Are samples transferred from the sampling device directly to their compatible containers? (Y/N) _____
2. Are sample containers for metals (inorganics) analyses polyethylene with polypropylene caps? (Y/N) _____
3. Are sample containers for organics analysis glass bottles with fluorocarbon resin-lined caps? (Y/N) _____
4. If glass bottles are used for metals samples, are the caps fluorocarbon resin-lined? (Y/N) _____
5. Are the sample containers for metals analyses cleaned using these sequential steps?
 - a. Nonphosphate detergent wash? (Y/N) _____
 - b. 1:1 Nitric acid rinse? (Y/N) _____
 - c. Tap water rinse? (Y/N) _____
 - d. 1:1 Hydrochloric acid rinse? (Y/N) _____
 - e. Tap water rinse? (Y/N) _____
 - f. Type II reagent grade water rinse? (Y/N) _____
6. Are the sample containers for organic analyses cleaned using these sequential steps?
 - a. Nonphosphate detergent/hot water wash? (Y/N) _____
 - b. Tap water rinse? (Y/N) _____
 - c. Distilled/deionized water rinse? (Y/N) _____
 - d. Acetone rinse? (Y/N) _____
 - e. Pesticide-grade hexane rinse? (Y/N) _____
7. Are trip blanks used for each sample container type to verify cleanliness? (Y/N) _____

B. Sample Preservation Procedures:

1. Are samples for the following analyses cooled to 4°C:
 - a. TOC? (Y/N) _____
 - b. TOX? (Y/N) _____
 - c. Chloride? (Y/N) _____
 - d. Phenols? (Y/N) _____
 - e. Sulfate? (Y/N) _____
 - f. Nitrate? (Y/N) _____
 - g. Pesticides/Herbicides? (Y/N) _____
 - h. Coliform bacteria? (Y/N) _____
 - i. Cyanide? (Y/N) _____
 - j. Oil and grease? (Y/N) _____
 - k. Volatile, semivolatile, and nonvolatile organics? (Y/N) _____

11. If samples are for inorganic analysis, does the cleaning procedure include the following sequential steps:
 - a. Nonphosphate detergent wash? (Y/N) _____
 - b. Dilute acid rinse (HNO₃ or HCl)? (Y/N) _____
 - c. Tap water rinse? (Y/N) _____
 - d. Type II reagent grade water? (Y/N) _____
12. If samples are for organic analysis, does the cleaning procedure include the following sequential steps:
 - a. Nonphosphate detergent wash? (Y/N) _____
 - b. Tap water rinse? (Y/N) _____
 - c. Distilled/deionized water rinse? (Y/N) _____
 - d. Acetone rinse? (Y/N) _____
 - e. Pesticide-grade hexane rinse? (Y/N) _____
13. Is sampling equipment thoroughly dry before use? (Y/N) _____
14. Are equipment blanks taken to ensure the sample cross-contamination has not occurred? (Y/N) _____

F. In-Situ or Field Analyses:

1. Are the following labile (chemically unstable) parameters determined in the field:
 - a. pH? (Y/N) _____
 - b. Temperature? (Y/N) _____
 - c. Specific conductivity? (Y/N) _____
 - d. Redox potential? (Y/N) _____
 - e. Chlorine? (Y/N) _____
 - f. Dissolved oxygen? (Y/N) _____
 - g. Turbidity? (Y/N) _____
 - h. Other (specify) _____
2. For in-situ determinations, are they made after well evacuation and sample removal? (Y/N) _____
3. If sample is withdrawn from the well, is parameter measured from a split portion? (Y/N) _____
4. Is monitoring equipment calibrated according to manufacturer's specifications and consistent with SW-846? (Y/N) _____
5. Is the date, procedure, and maintenance for equipment calibration documented in the field logbook? (Y/N) _____

C. Sampling of Immiscible Layers:

1. Are the immiscible layers sampled separately prior to well evacuation? (Y/N) _____
2. Do the procedures used minimize mixing with water soluble phase? (Y/N) _____

D. Water Evacuation:

1. Are low yielding wells evacuated to dryness? (Y/N) _____
2. Are high yielding wells evacuated so that at least three casing volumes are removed? (Y/N) _____
3. What device is used to evacuate the wells? _____
4. If any problems are encountered (e.g., equipment malfunction) are they noted in a field logbook? (Y/N) _____

E. Sample Withdrawal:

1. For low-yielding wells, are first samples tested for pH, temperature, and specific conductance after the well recovers? (Y/N) _____
2. Are samples collected and containerized in order of the parameters volatilization sensitivity? (Y/N) _____
3. For higher-yielding wells, are samples retested for pH, temperature, and specific conductance to determine purging efficiency? (Y/N) _____
4. Are samples withdrawn with either fluorocarbon resins or stainless steel (304, 316, 2205) sampling devices? (Y/N) _____
5. Are sampling devices either bottom valve bailers or positive gas displacement bladder pumps? (Y/N) _____
6. If bailers are used, is fluorocarbon resin-coated wire, single strand stainless steel wire, or monofilament used to raise and lower the bailer? (Y/N) _____
7. If bailers are used, are they lowered slowly to prevent degassing of the water? (Y/N) _____
8. If bailers are used, are the contents transferred to the sample container in a way that will minimize agitation and aeration? (Y/N) _____
9. Is care taken to avoid placing clean sampling equipment on the ground or other contaminated surfaces prior to insertion into the well? (Y/N) _____
10. If dedicated sampling equipment is not used, is equipment disassembled and thoroughly cleaned between samples? (Y/N) _____

FIELD AUDIT CHECKLIST

Project No.: _____

Sampling Date: _____

Description of Project: _____

Name of Sampler(s): _____

Checklist Completed By: _____

I. REVIEW OF SAMPLE COLLECTION PROCEDURES

A. Measurement of Well Depths Elevation:

1. Are measurements of both depth to standing water and depth to the bottom of the well made? (Y/N) _____
2. Are measurements taken to the nearest centimeter or 0.01 foot? (Y/N) _____
3. What device is used? _____
4. Is there a reference point(s) established by a licensed surveyor? (Y/N) _____

B. Detection of Immiscible Layers:

1. Are procedures used which will detect light phase immiscible layers? (Y/N) _____
2. Are procedures used which will detect dense phase immiscible layers? (Y/N) _____

CORNING DISPOSABLE FILTER COMPONENTS

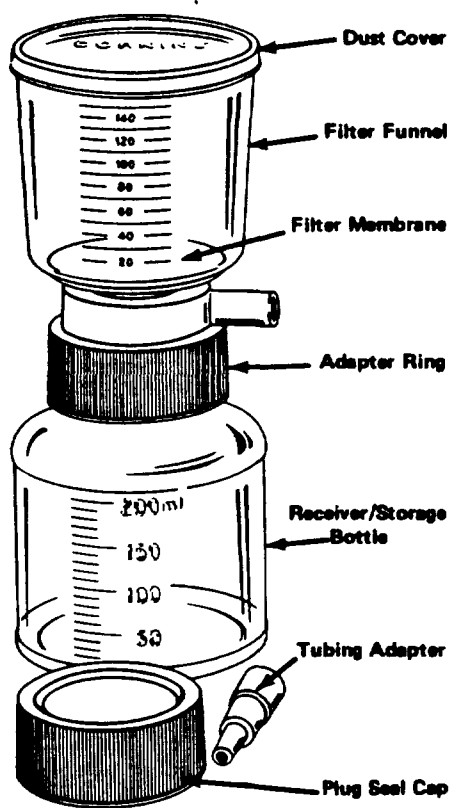


Figure 1.

- b. Filter membrane must be covered completely by prefilter to work properly.
 - c. Prefilter must be placed rough side up to work.
3. Attach vacuum pump to filter; turn on power.
 4. Filter groundwater sample until appropriate amount of sample needed is filtered.
- NOTE: More filters may be needed to get an appropriate sample volume.
5. After filtering is complete, pour contents into the appropriate sample container, dispose of filter (never reuse filters).

DOCUMENTATION:

The technicians will document the number of filters and prefilters used for each sample filtered on the field log data sheet.

**STANDARD OPERATING PROCEDURES
FOR THE
FILTERING OF GROUNDWATER SAMPLES FOR METALS**

PURPOSE: Description of the filtering process for groundwater samples.

APPLICABILITY: These procedures apply to the filtering of groundwater for laboratory analysis.

REFERENCES: Corning Disposable Sterile Filter Information Booklet.

DISCUSSION: Filtering is done on groundwater samples to remove silt, clay, etc.

RESPONSIBILITIES: The environmental technicians are responsible for the filtering of groundwater samples.

PROCEDURE: Filtering Process:

1. Collect groundwater sample in an unpreserved sample container (filtering should be done within 15 minutes of collection).
2. Pour groundwater sample into 200-ml or 500-ml Corning Disposable Sterile Filter (see Figure 1), depending on volume needed.
3. The filters must be 0.45 micron pore size.

NOTE: Prefiltering may be needed if sample is too turbid. The prefilter will filter particles up to 0.60 micron pore size.

- a. Add prefilter to filter by placing it over the filter membrane (extends the life of the filter).

2. Half fill a clean glass jar with the sample to be analyzed using a stainless steel spoon. Quickly cover the open top of the jar lid on tightly to seal the jar.
3. Vigorously shake the jar for 15 seconds.
4. Allow headspace development for 10 minutes. Ambient temperature during headspace development should be recorded. When ambient temperatures are below 32°F, headspace development should be conducted inside a heated vehicle or building.
5. Vigorously shake the jar for an additional 15 seconds.
6. Remove the jar lid to expose the aluminum foil seal. Quickly puncture the foil seal with the sampling probe to a point about one-half of the headspace depth. Exercise care to avoid uptake of water droplets or soil particles.
7. Record the highest meter response as the headspace concentration. The maximum response will likely occur between two to five seconds.

<u>Oil Sheen Test Result</u>	<u>Description</u>
None	No sheen detected.
Trace	Possible or faint oil sheen observed (May not continue to generate sheen as additional water is added).
Moderate	Definite oil sheen, but "rainbow colors" not distinguishable.
Heavy	Definite oil sheen with "rainbow colors" observed.

Interferences on the test can be caused by any contaminant which will cause an oil sheen on water. The samples will be carefully observed for characteristic appearance or odors which may indicate a possible contaminant other than coal tar.

Odor. Odor will be described as low, moderate, strong, or very strong coal tar odor, or as diesel or petroleum odor. The sampler will note odor only if noticed incidentally while handling the soil sample. The sampler will not place themselves at risk.

Headspace Organic Vapor Screening. The headspace organic vapor screening method will be used in the field to screen soils for organic vapors. The screening method is intended to be used in conjunction with other "real time" observations which include a description of the odor and appearance of the soil sample and an measure of the oiliness of the soil sample.

The equipment required to conduct headspace organic vapor screening includes: a clean pint or quart-size glass jar with lid, aluminum foil, stainless steel spoon, a log book or recording sheet, and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the Health and Safety Plan.

The following procedure will be used for conducting headspace organic vapor screening:

1. Soil samples collected from a split-barrel sampler will be collected immediately after opening the split-barrel. If the sample is collected from an excavation wall, soil pile, or backhoe bucket, it will be collected from a freshly exposed surface.

STANDARD OPERATING PROCEDURE
FOR
FIELD SCREENING TECHNIQUES
FOR
SOILS CONTAINING COAL TAR

The field screening techniques for soils containing coal tar are as follows: (1) Visual Examination; (2) Oil Sheen; (3) Odor; and (4) Headspace Organic Vapor Screening. The results of these four screening procedures will be used to determine the gross level of PAH contamination of the soil sample.

Visual Examination. A visual examination of the soil sample will include noting any discoloration of the soil or the presence of coal tar.

Oil Sheen Test. The oil sheen test is a method used to immediately determine the approximate magnitude of coal tar contamination in soil by observation of the sample in the field. The test is useful in soils which do not have a high binding capacity with polyaromatic hydrocarbons (PAHs) (i.e., the PAHs are free on the surface of the soil particles and can be released by a stream of water).

The equipment required to conduct the oil sheen test includes: a stainless steel spoon, a squirt bottle filled with tap water, a log book or recording sheet, and the appropriate personal protective equipment necessary for collection and handling of soil samples as described in the Health and Safety Plan. Decontamination of the spoon between test events will consist of scrubbing the surface of the spoon with a solution of trisodium phosphate in water using a brush and then rinsing the spoon with water.

The procedure for conducting the oil sheen test consists of obtaining approximately 50 grams (about 30 cc) of representative soil with the spoon and then directing a stream of water onto the soil in the spoon with the squirt bottle until the soil is saturated and water begins to collect around the soil. The amount of oil sheen present on the water is determined by observation and the results of the test are reported as a magnitude of oil sheen observed: none, trace, moderate, or heavy. The test results, sample location, and observations of the sample's appearance and odor are recorded in the log book.

The specific soil types at the area of investigation should be accounted for when performing the oil sheen test. The best results are obtained in silts, sands, and/or gravels with low organic content. The results obtained from clayey soils may appear deceptively low. Typical descriptions of each test result are given below.

5. Check pH electrode connection to meter.
6. Clean pH electrode and replace reference solution.
7. Use fresh buffer solutions.
8. Try new probe.
9. Send in meter to be repaired.

Sample analysis:

1. Rinse pH electrode.
2. Place electrode into sample.
3. Wait for pH reading to stabilize (1 to 5 minutes).
4. Read and record pH reading to the nearest tenth unit.
5. Remove electrode from sample and rinse.
6. Store electrode in buffer solution or storage solution between sample measurements.

QUALITY CONTROL:

Accuracy of field measurement of pH will be determined by calibration verifications every five samples collected and at the end of the day. The accuracy will be assessed by performing two measurements on two standard buffer solutions which bracket the pH range of the samples. Each measurement will be within ± 0.1 standard unit of buffer solution or the meter will be recalibrated.

Precision will be assessed through duplicate measurements at a frequency of 10 percent or one per day minimum. If duplicate measurement of pH is not within 0.1 pH units, the pH meter will be recalibrated.

DOCUMENTATION:

The technician will document the calibration and any pertinent information in each meter's log book. Calibration will be done at the start of the sampling day. Calibration verification will be done after every five samples collected and again at the end of the day. pH values of samples will be written down on the field log data sheets for each sample collected.

STANDARD OPERATING PROCEDURES
FOR THE
CALIBRATION AND OPERATION
OF THE pH METER

PURPOSE: The purpose is to describe the use of the pH meter, its calibration, documentation and readings.

RESPONSIBILITIES: The field technicians are responsible for the use, calibration, and documentation of pH readings.

EQUIPMENT:	Orion Model 407A/F	Reads pH 0.1 units
	Orion Model SA205	Reads pH 0.1 or 0.01 units
	Orion Model pH60	Reads pH 0.01 units

PROCEDURES: Two-buffer calibration:

1. Turn meter on, let warm up three minutes.
2. Connect pH electrode to meter.
3. Place meter in pH mode.
4. Place electrode into calibration solution #1 (7.00 buffer).
5. Let reading stabilize, adjust reading to 7.00, if necessary.
6. Rinse electrode, place into calibration solution #2 (10.00 buffer).
7. Let reading stabilize, adjust reading to 10.00.
8. Rinse electrode and place into sample.
9. Read pH off of meter to nearest tenth.

Accuracy requirements:

A properly functioning electrode and meter will have a slope of 90 percent to 102 percent. The slope is checked after calibration using the following procedure:

1. Place meter in slope mode.
2. Read slope.
3. If in range, proceed to take reading; if out of range, these steps are necessary.
4. Check battery, replace or recharge if low.

NOTE: The conductivity model, the user must set dial to desired range of measurement (X1, X10, X100 umhos/cm range) according to the sample conductivity.

3. Place probe into sample and move it around in the sample to remove any air bubbles inside the probe.
4. Wait for measurement (temperature or conductivity) to stabilize (about 1 to 5 minutes).
5. Read and record conductivity measurement and temperature.

NOTE: Conductivity must be calculated to the standardized 25°F.

DOCUMENTATION:

The technicians will document the procedures done in redlining daily and checking of conductivity weekly. This will be written down in the log book for each meter. Any other pertinent information will also be noted in the log book for each meter. The conductivity and temperature readings for each sample will be recorded on the field log data sheets for each sample collected.

Checking Calibration of Conductivity (once per week)

1. Redline meter.
2. Put probe into conductivity calibration solution (conductivity calibration solution has a fixed conductivity at a specific temperature established by the manufacturer; YSI 3167 conductivity calibrator assayed at 0.997 millimho/cm (997 micromho/cm), contains potassium chloride, water, and 0.002% iodine).
3. Check temperature of solution.
4. Check conductivity of solution.
5. Match meter readings with prescribe readings (prescribe readings are found accompanying the solution).
6. Reading should be within 50 umhos/cm of the prescribe reading.
4. If conductivity readings do not match:
 - a. Check probe connection to meter
 - b. Change battery
 - c. Clean probe
 - d. Try different conductivity solution
 - e. Replatinize probe
 - f. Change probe
 - g. Send meter to be fixed

Sample Analysis:

1. Rinse conductivity probe.
2. Select desired mode (temperature or conductivity).

STANDARD OPERATING PROCEDURES
FOR THE
CALIBRATION AND OPERATION
OF THE
CONDUCTIVITY AND TEMPERATURE METER

PURPOSE: The purpose is to describe the use of the conductivity and temperature meter, its calibration, documentation and readings.

APPLICABILITY: These procedures apply to finding the conductivity and temperature of a water sample.

DEFINITIONS: Redline: This is a setting on the conductivity and temperature meter to show if the meter is zeroed and to check the battery.

REFERENCE: The Instructions for YSI Model 33 S-C-T Meter, Yellow Springs Instrument Co., Yellow Springs, Ohio.

RESPONSIBILITIES: The Environmental Technicians are responsible for the operation, maintenance, and checking calibration of conductivity and temperature meter.

PROCEDURE: Calibration (Daily Verification)

1. Turn meter to redline.
2. Meter warm up (2 to 3 minutes).
3. The meter needle should then be exactly on the redline (located on the far right of the display screen).
4. Adjust the redline dial accordingly to receive and accurate display.
5. If redline malfunctions change battery.

Appendix A

Barr Engineering Co. Standard Operating Procedures

FIGURE 15.1-1
CORRECTIVE ACTION FORM

Project No.: _____ Date: _____
Project Name: _____ Initiated by: _____
Project Manager Notified: _____

Problem Description:

Corrective Action Taken:

Project Manager Informed: _____
Date: _____

Figure 14.1-1

Project #: _____
Date: _____
Reviewed by: _____

QUALITY CONTROL REVIEW

1. Holding Time Met: yes _____
no _____

2. Accuracy Data:

3. Precision Data:

4. Masked Duplicate Analysis:

5. Standards Data:

6. Blank Results:

7. Comparison with Previous Data:

Comments:

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Rev. No. 3
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- 6.2.1 Rinse a 5-ml gas-tight syringe with deionized water three times, once with methanol, and once with reagent water.
- 6.2.2 Cap the end of the syringe with a stop-go valve and add reagent water to just short of overflowing. Insert the plunger, invert the syringe(plunger side down), press the plunger to remove air bubbles and excess water. Final volume in the syringe should be 5 ml.
- 6.2.3 Add calibration standards, surrogate standards, and internal standards to the valve end of the syringe per the following table. Use syringes specifically designed for each of these solutions.

Working Solutions

Description	Conc. (ug/ml)	Vol. to use (ul)	Final Conc. (ug/ml)
Calibration Standards	25	1	5
		4	20
		8	40
		20	100
		40	200
Surrogate Standards	25	1	5
		4	20
		8	40
		20	100
		40	200
Internal Standards	25	8	40

These volumes are to be used for preparing the initial calibration curves for a 5 ml purge volume. When 25 ml are to be purged, spike the reagent water with five times(5x) the above indicated amounts.

- 6.3³ Analyze each calibration standard and calculate the relative response factor (RRF) for each of the compounds according to the following equation:

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$$RRF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

Where:

A_s = Area of Analyte

A_{is} = Area of internal standard

C_{is} = Concentration of internal standard

C_s = Concentration of analyte

NOTE: Certain data processors may calculate the RRF differently.

- 6.4 Calculate the standard deviation (S) and the relative standard deviation (%RSD) of RRFs for the compounds using the following equations:

$$S = \sqrt{\frac{\sum (RRF_i - RRF_m)^2}{N-1}} \sqrt{\frac{1}{2}}$$

RRF_i = Individual RRF

RRF_m = Mean RRF

N = Number of RRFs

$$\text{and } \%RSD = \frac{S \times 100}{RRF_m}$$

- 6.5 The Relative standard deviation of each compound must be less than 30 percent. This criteria must be achieved for the calibration to be valid.
- 6.6 If the relative standard deviation is less than 20 percent, the RRF of the compound can be assumed to be invariant, and the average RRF can be used for calculations.

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6.7 If the relative standard deviation is between 20% and 30 percent, calculations must be made from the calibration curve. Both the slope and the intercept of the curve must be used to perform calculations.

6.8 The validity of the calibration curve must be validated further by the analysis of a QC Check sample. The QC check sample must be obtained from EPA, another vendor, or it must be from another lot number. The QC check sample verifies the validity of the concentrations of the standards used to obtain the initial calibration.

Prepare and analyze a midpoint aqueous QC check standard. For each parameter, calculate the percent recovery. All parameters in the QC check standard must be recovered within 70 - 100 percent. If any parameter exceeds this criterion, then a new calibration curve must be established. All sample results for a target analyte can only be reported from valid initial calibrations.

7.0 Continuing Calibration

The working calibration curve or relative response factor for each analyte must be verified daily by the analysis of a continuing calibration standard. The ongoing daily continuing calibration must be compared to the initial calibration curve to verify that the operation of the measurement system is in control.

7.1 Continuing Calibration - The frequency of the continuing calibration check must be performed for each day of analysis to verify the continuing calibration of the instrument. A day is defined as 12 hours from the start run time of the last valid continuing calibration.

A 20 ppb daily continuing calibration is prepared with all target compounds present(see sec.6.2).

7.2 Verification of continuing calibration is performed by the analysis of a midpoint standard containing all of the parameters of interest. Verification of continuing

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calibration of the measurement system can be performed in one of the following two ways:

- 7.2.1 Analyze the midpoint continuing calibration standard and calculate the relative response factor (RRF) for each of the target analyte. For each analyte, calculate the percent difference of the continuing calibration RRF from the mean RRF from the initial calibration curve using the following equation:

$$\%D = \left[\frac{RRF_m - RRF}{RRF_m} \right] \times 100$$

Where:

RRF_m = The mean relative response factor from the initial calibration curve.

RRF = The daily relative response factor.

The criteria for acceptable continuing calibration is 15% D. No more than four analytes for method 8010 and two analytes for method 8020 can exceed the acceptance limits. (NOTE: If one of the target analytes that exceeded the 15% D criteria in detected in a sample, then a new calibration of the instrument must be performed for that target analyte, and the sample must be re-analyzed after a valid initial calibration is achieved). If the acceptance criteria are achieved, the calibration curve can be used to calculate the results of sample analyses. If the criteria are not achieved, another new calibration curve must be prepared.

An "Average Response Factor" for each compound in the 5-point curve is generated. Daily Response Factors are calculated for each compound in the Continuing Calibration. The "% Difference" between the two response factors is calculated. The acceptable limits for % D for each compound were computed using Table 2 Values in Methods 602 and 8020. For those compounds that are not on the 602 or 8020 target lists (TBME, Xylenes), a limit of 50% is used. The SOP allows up to two compounds to

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have % D values exceeding the limits in a continuing calibration, but if there are any target hits for those two compounds, the sample must be rerun against a Continuing Calibration that meets % D criteria for those compounds. Copies of two recent Continuing Calibration Forms are in Attachment A.

- 7.2.2 Analyze the midpoint continuing calibration standard and calculate the relative response factors (RRFs) of each of the target analytes. If no more than four 8010 and two 8020 compounds exceed the 15% D criteria, then the continuing calibration response factors can be updated, and the sample results can be calculated from the updated response factor. (NOTE: If one of the target analytes that exceeded the 15% D criteria is detected in a sample, then a new calibration of the instrument must be performed for that target analyte, and the sample must be re-analyzed after a valid initial calibration is achieved). If these criteria are exceeded, a new calibration curve must be prepared.

All sample results for any target analyte can only be reported from a valid initial and continuing calibration.

8.0 Quality Control

- 8.1 Method Blanks - The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by the analysis of method blanks. Each day before the analysis of samples, an organic free water blank must be analyzed to demonstrate the system is clean.

Contamination by carry-over can also occur when high level and low level samples are analyzed in sequence. To reduce the possibility of carryover, the purging device and sample syringe must be rinsed with organic free water between samples. Also, frequent bake-out and purging of the entire system may be required. For samples containing high concentrations of any volatile analyte, an organic free water blank should be analyzed immediately afterwards to verify that the analytical system is clean before the analysis of other samples. If samples are analyzed by an autosampler, all sample

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analytical results following a high level sample must be examined for the possibility of crossover. If target analytes are found in the samples that are common to a previously analyzed high sample, the samples must be re-analyzed after demonstration of a clean analytical system by analysis of blanks.

The blank analyzed for the high level soil methanol extraction method must include the same amount of the reagent methanol that is added during the analysis of the samples.

The results of blank analyses must be reported along with the results for samples. The blank results should not be subtracted from the sample analytical results.

A method blank must contain no greater than five times (5x) the stated method detection limit of common laboratory solvents such as methylene chloride. For all other volatile compounds, the method blank must contain less than the stated detection limit. If the method blank exceed these criteria, the laboratory must consider the analytical system out of control. Appropriate corrective actions must be taken and documented before further sample analysis.

8.2 QC Check Samples - The validity of the calibration curve must be verified by the analysis of a QC check sample that has been obtained from another source before the analysis of any samples. The QC check sample verifies the validity of the concentrations of the standards used to prepare the calibration curve. The QC check sample must be obtained from EPA, another vendor or from another lot number. The initial calibration curve is valid if all target analytes are within 70 - 130 percent recovery. After the curve is verified from independent standards, continuing calibration of the system can be verified daily from the midpoint standard that was used to prepare the initial calibration curve.

8.3 Matrix Spikes/Matrix Spike Duplicates (MS/MSD) -At least one set of MS/MSD samples must be analyzed for each 20 samples to acquire data for measurement of accuracy and precision.

The working calibration solution(see sec. 4.4.1) includes all the target analytes for the method. The percent

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recoveries of the target analytes are compared to the acceptable range of recoveries given in Tables 3 and 4. If more than four target analytes for method 601 and two target analytes for method 602 exceed the acceptance criteria, a continuing calibration check sample must be analyzed to check instrument calibration. If continuing calibration criteria are not achieved for the parameters that failed the limits for spike recovery, the affected samples must be re-analyzed using a new calibration curve. If continuing calibration criteria are achieved ($\pm 15\%D$), the poor recoveries can be attributed to a matrix effect. The results from both sample analyses must be reported.

8.3.1 Matrix Spike Solution - Prepare the MS/MSD as for the unspiked sample, adding in addition to the surrogates and internal standard, 4 ul of the working calibration solution(sec. 4.4.1).

8.4 Surrogate Spike Results - All samples and blanks must be spiked with surrogate compounds prior to purging. Data for the surrogates for all analyses must be tabulated and routinely processed statistically for determination of warning and control limits. The acceptable range for surrogate recovery is 65% - 135%. If a recovery falls outside of these limits, the sample will be reanalyzed unless there is obvious surrogate interference. If the recovery again falls outside the limits, both analytical runs will be reported with a corresponding narrative.

8.4.1 We currently use A,A,A-Trifluorotoluene as the surrogate at a 50 ppb concentration. A 10 ul aliquot of the surrogate working solution(sec. 4.4.2) is added to a 5 ml volume, resulting in a 50 ppb concentration. 4-Bromofluorobenzene is used for the internal standard at a 20 ppb concentration. A 4 ul aliquot of the internal standard working solution(sec. 4.4.4) is added to a 5 ml volume, resulting in a 20 ppb concentration. When a 25 ml volume is purged, a 50 ul aliquot of surrogate solution, and a 20 ul aliquot of internal standard solution are used.

9.0 Sample Analysis

9.1 Continuing Calibration Check - Each day before the analysis of samples, prepare and analyze a midpoint water

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standard according to directions given in section 6. The calibration analysis should be performed according to the method used to analyze samples as discussed in section 6.1. Verification of continuing calibration must be checked against a calibration curve that was determined using the same sample volumes and instrument conditions.

- 9.2 Method Blank - Analyze a method blank to verify that the analytical system is free of contamination under the conditions of analysis. Use the same sample volumes, reagents, and instrument conditions used for the samples.
- 9.3 Analysis of Water Samples - Allow all samples and standard solutions to come to ambient temperature before analysis. Adjust the purge gas flow rate to 40 mL/min. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 or 25.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data.

If dilution of the sample is needed, the dilutions should be made with volumetric flasks and the sample syringe. **NOTE: Pipettes should not be used to measure aliquots of water samples for volatiles.**

For an undiluted sample, 5 ml is the amount injected. Dilutions may be necessary to keep the results within linear range. Dilutions may also be necessary if non-target compounds are present in the matrix that would overload or contaminate the system. As mentioned, all soil samples are screened. All water samples are screened as well, unless historical data is sufficient to provide necessary dilutions.

Add the surrogate and internal standard spiking solution through the valve bore of the syringe, and close the valve.

Inject the sample into the purging device, and purge for 11 minutes at ambient temperature. At the conclusion of the purge time, adjust the purge and trap device to the

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desorb mode and begin the temperature program of the gas chromatograph. While the trap is being desorbed, wash the purging chamber with two 5-mL aliquots of blank water. After desorbing the sample for four minutes, recondition the trap by adjusting the device to the "bake" mode. The trap temperature should be baked at 180°C for at least 7 minutes. Allow the trap to cool before the analysis of the next sample.

All target analytes detected must be within the linear calibration range established for the instrument. If a target analyte exceeds the calibration range, the sample must be diluted in such a manner that response of the major constituents are in the upper half of the calibration range.

For water samples, add the matrix spike solution to the 5 mL of sample purged.

9.4 Analysis Of Sediment/Soil Samples - Sediment or soil samples are analyzed in one of two ways depending on the concentration of analytes in the sample.

Low-level soils are analyzed using a heated water bath, and Methanol extracts are made from medium-level soils. Extracts are made of all soil samples and "screened" on an FID before analysis. The screening procedure helps to identify medium-level soils and aids in determining the final dilution needed. The low level method of analysis involves the analysis of a 1 to 5 gram aliquot of the sample suspended in water using the heated purge technique. The smallest sample size permitted for this analysis is 1 gram. If smaller than 1 gram sample size is needed to stay within the linear calibration range, then the medium level method must be used. The medium level method involves extracting the soil sample with methanol, spiking an aliquot of the extract into blank water, and analyzing the spiked water in the same manner as a water sample.

9.4.1 Low Level Method - The low level method is performed by purging a suspension of the matrix in water. The suspension is heated to 40°C during the purging process. All standards, blanks, and samples must analyze under the same conditions.

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Mix the contents of the sample well, and weigh a 1 to 5-gram aliquot of the solid matrix into a tared purge device. The sample should be weighed on a top loading balance to the nearest 0.1 gram. Spike the appropriate amount of the internal standard and surrogate spiking solution to a 5-mL aliquot of blank water(sec. 8.4.1). Add the spiked blank water to the purge device, and connect the device to the purge and trap system. Heat the sample to 40°C and purge the sample for 11 minutes. Proceed with the analysis as described for water analysis.

For low level sediment/soils, add the matrix spike solution to the 5-mL of water and soil mixture.

- 9.4.2 Medium Level Method - If the analysis of less than 1 gram of a solid sample is indicated, then the medium level method must be used for analysis. The medium level method is based on the analysis of a methanol extract of the soil matrix. An aliquot of the methanol extract is spiked into blank water, and purged at ambient temperature as prescribed for water samples.

Mix the contents of the sample container well, weigh 5 grams of the matrix into a tared 40 ml vial. Using a toploading balance, weigh the sample to the nearest 0.1 gram. Quickly add 5.0 mL of methanol to the vial. Cap the vial and shake for 2 minutes. This extraction procedure must be performed in a laboratory free of solvent fumes.

Pipette approximately 1 ml of the methanol extract into a GC vial. Also, transfer approximately 1 ml of the reagent methanol used to perform the extraction to a 1-mL GC vial. These extracts should be stored in the dark at 4°C until the analysis.

The quantity of the extract that is spiked into the blank water is determined by prior screening or analysis results. The maximum amount of methanol should not exceed 100 uL per 5 ml of spike water. It is important that the volume of the methanol remain the same for all samples, blanks, and standards. If less

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than 100 uL is needed for proper dilution, then add additional reagent methanol as necessary to maintain a total volume of 100 uL added to the syringe.

Remove the plunger from a 5-mL syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL and pull the plunger back from the 5-mL mark to allow volume for the addition of sample and standards. Add the internal standard and surrogate standard solution. Add the methanol extract and an appropriate volume of reagent methanol to maintain a total volume of 100 uL.

Inject the sample into the purge device and analyze according to procedures given for water analysis.

For the medium level method, add the matrix spiking solution to the syringe as with water samples. The total volume of methanol added to the 5 mL of water must be 100 uL.

10. Data Calculation, Review, Validation and Reporting

10.1 Data Calculation - Calculate the concentration of target analytes in the sample using the following equation:

NOTE: The following equations can only be used if the instrument calculates the RRFs according to the equation given in section 6.4.

10.1.1 Water

$$\text{Concentration (ug/L)} = \frac{A_x \times I_s}{A_{is} \times \text{RRF} \times V_o}$$

Where:

A_x = Area for the compound to be measured.
A_{is} = Area internal standard.
I_s = Nanograms of internal standard added.
V_o = Volume of water purged mLs - Take into account any dilutions.

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10.1.2 Low Level Soil/Sediment

$$\text{Concentration (ug/kg)} = \frac{Ax \times Is}{Ais \times RRF \times D \times Ws}$$

Where:

Ax = Area for the compound to be measured.
Ais = Area for the specified internal standard.
Is = Nanograms of internal standard added
D = $\frac{100 - \% \text{ Moisture}}{100}$
Ws = Grams of solid extracted

10.1.3 Medium Level Soil/Sediment

$$\text{Concentration (ug/kg)} = \frac{Ax \times Is \times Vt}{Ais \times RRF \times Vi \times D \times Ws}$$

Where:

Ax = Area for the compound to be measured.
Ais = Area for the internal standard.
Is = Nanograms of internal standard added
D = $\frac{100 - \% \text{ Moisture}}{100}$
Vt = Volume of total extract (mLs).
Vi = Volume of extract added for purging (mLs)
Ws = Grams of soil extracted

10.2 Data Review and Validation - The data acquired for a sample must be reviewed and validated against the quality control data acquired during the analysis. The following list of items must be checked and meet acceptance criteria before approval for reporting:

- * Valid initial calibration
- * Valid QC Check Sample
- * Valid continuing calibration
- * Method blank that meets criteria in Section 8.1.
- * Matrix spike that meets criteria of Section 8.3.
- * Surrogates must meet criteria of Section 8.4.
- * The results of all samples that follow a high sample that has the potential for crossover must be examined carefully for the possibility of crossover.
- * All dilutions accounted for in the calculation.

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- * The relative retention time of an identified analyte must be within $\pm 0.0.1$ (0.02 Packed) of the relative retention time of the daily standard.
- * Whenever questions of identity exists, the target analyte found in the samples by GC analysis should be confirmed by another column, GC/MS analysis, or response factor ratio comparison if response is generated by both detectors.

10.3 Data Reporting - Before reporting the data for a sample, the data should be reviewed by a senior level analyst that is not involved in the actual analysis. The report should be reviewed for transcription errors, and calculations should be spot-checked.

The reporting limits must be reported with correction for any extract dilution and the percent solids. The following equation must be used to correct the reporting limits for dilution and percent solids:

If the nominal reporting limit for soil is 5 ug/kg, and one gram was purged, and the percent solids is 50%, then correction is made as follows:

$$\frac{(5) \times DF}{D} \quad \text{or} \quad \frac{5 \times 5}{0.5}$$

The associated QC data for blanks and surrogates must be reported with the sample data. The values obtained for common laboratory contaminants such as methylene chloride should be reported for both blanks and samples without blank subtraction.

Any unusual difficulties experienced with the sample analyses should be documented and noted in the analytical report.

11. Documentation of Quality Control Data

All quality control data generated using these methods must be separated from the sample data files and bound in three ring binders for easy access and review. It is recommended that a separate three ring binder be maintained for each instrument used for these analyses.

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The three ring binder should be subdivided into the following sections:

- * Initial Calibration Results
- * QC Check Results
- * Continuing Calibration Results
- * Method Blank Results
- * MS/MSD Results
- * Surrogate Spike Results and Control Charts

The data should be stored in order of data acquired. In this manner, all sample data can be easily referenced to the associated QC data.

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TABLE 1

Method 8010 Halocarbons and Method Detection Limits

601 Compounds	MDL (ug/L)	MDL (ug/L)
	5 mL Sample	25 mL Sample
Chloromethane	1.0	0.5
Bromomethane	1.0	0.5
Dichlorodifluoromethane	1.0	0.5
Vinyl Chloride	1.0	0.5
Chloroethane	1.0	0.5
Methylene Chloride	1.0	0.5
Trichlorofluoromethane	1.0	0.5
1,1-Dichloroethene	1.0	0.5
1,1-Dichloroethane	1.0	0.5
trans-1,2-Dichloroethane	1.0	0.5
Chloroform	1.0	0.5
1,2-Trichloroethane	1.0	0.5
Carbon tetrachloride	1.0	0.5
Bromodichloromethane	1.0	0.5
1,2-Dichloropropane	1.0	0.5
cis-1,3-Dichloropropene	1.0	0.5
Trichloroethene	1.0	0.5
Dibromochloromethane	1.0	0.5
1,1,2-Trichloroethane	1.0	0.5
trans-1,3-Dichloropropene	1.0	0.5
Bromoform	1.0	0.5
1,1,2,2-Tetrachloroethane	1.0	0.5
Tetrachloroethene	1.0	0.5
Chlorobenzene	1.0	0.5
1,3-Dichlorobenzene	1.0	0.5
1,2-Dichlorobenzene	1.0	0.5
1,4-Dichlorobenzene	1.0	0.5

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TABLE 2

Method 8020 Halocarbons and Method Detection Limits

602 Compounds	MDL (ug/L)	MDL (ug/L)
	<u>5 mL Sample</u>	<u>25 mL Sample</u>
Benzene	1.0	0.5
Chlorobenzene	1.0	0.5
Toluene	1.0	0.5
Ethylbenzene	1.0	0.5
Chlorobenzene	1.0	0.5
1,3-Dichlorobenzene	1.0	0.5
1,2-Dichlorobenzene	1.0	0.5
1,4-Dichlorobenzene	1.0	0.5
Xylene	1.0	0.5

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TABLE 3
 EPA 601 MATRIX SPIKE ACCEPTANCE CRITERIA

Compound	Percent Recovery	Acceptable Recovery Range 1
Chloromethane		D-193
Bromomethane		D-144
Dichlorodifluoromethane		*
Vinyl Chloride		28-163
Chloroethane		46-137
Methylene Chloride		25-162
Trichlorofluoromethane		21-156
1,1-Dichloroethene		28-167
1,1-Dichloroethane		47-132
trans-1,2-Dichloroethene		38-155
Chloroform		49-133
1,2-Dichloroethane		51-147
1,1,1-Trichloroethane		41-138
Carbon tetrachloride		43-143
Bromodichloromethane		42-172
1,2-Dichloropropane		44-156
cis-1,3-Dichloropropene		22-178
Trichloroethene		35-146
Dibromochloromethane		24-191
1,1,2-Trichloroethane		39-136
trans-1,3-Dichloropropene		22-178
Bromoform		13-159
1,1,2,2-Tetrachloroethane		8-184
Tetrachloroethene		26-162
Chlorobenzene		38-150
1,3-Dichlorobenzene		7-187
1,2-Dichlorobenzene		D-208
1,4-Dichlorobenzene		42-143

1 Reference: EPA Method 8010, Table 3, Test Methods for Evaluating Solid Waste, SW-846 Manual, November 1986.

* Criteria not listed.

TABLE 1

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TABLE 4
EPA 602 MATRIX SPIKE ACCEPTANCE CRITERIA

Compound	Percent Recovery	Acceptable Recovery Range 1
Benzene		39-150
Toluene		46-148
Ethylbenzene		32-160
Chlorobenzene		55-135
1,4-Dichlorobenzene		42-143
1,3-Dichlorobenzene		50-141
1,2-Dichlorobenzene		37-154
Xylenes		40-150

1 Reference: EPA Method 8010, Table 3, Test Methods for Evaluating Solid Waste, SW-846 Manual, November 1986.

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ATTACHMENT A

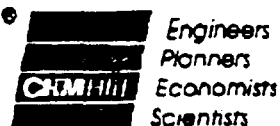


Engineers
Planners
Economists
Scientists

VQA INITIAL CALIBRATION FORM
GC81/DB-624 MEGABORE CAPILLARY
STANDARD: CHEN SERV
FILE NAME: 11C0119A
DATE: 1/19/91

COMPOUND NAME	RF 5 PPS	RF 20 PPS	RF 40 PPS	RF 100 PPS	RF 200 PPS	AVERAGE RF	STD DEV (N-1)	% RSD	XRSD MUST BE <30 USE AV RF IF XRSD <20
DCDFH	10.5412	8.8444	8.1434	8.6456	7.8462	8.8038	1.0491	11.9164	
CH3CL	2.6467	2.2488	2.1933	2.3863	2.2500	2.3451	0.1830	7.8038	
VINYL CL	2.0016	1.7702	1.7283	1.9139	1.8173	1.8463	0.1110	6.0097	
CH3BR	2.2325	1.9367	1.8851	2.0422	1.8917	1.9980	0.1453	7.2746	
CL ETHANE	1.1212	1.1289	1.1719	1.3205	1.3152	1.2115	0.0990	8.1689	
TCFH	0.8504	0.8493	0.8490	0.9871	0.8776	0.8827	0.0596	6.7564	
1,1-DCE=	0.5753	0.4734	0.5163	0.6115	0.5580	0.5469	0.0535	9.7792	
DCM	0.4881	0.4497	0.3905	0.4860	0.4627	0.4514	0.0398	8.8137	
1-12-DCE=	0.3568	0.3450	0.3472	0.4152	0.4131	0.3733	0.0356	9.4829	
1,1-DCE	0.3764	0.3804	0.4051	0.4916	0.4204	0.4148	0.0466	11.2319	
CHCL3	0.3167	0.2726	0.3056	0.3327	0.3409	0.3133	0.0267	8.5286	
1,1,1-TCE	0.4482	0.3705	0.4054	0.4717	0.4552	0.4302	0.0416	9.6196	
CCL4	0.3274	0.3183	0.3419	0.3871	0.3801	0.3510	0.0311	8.8502	
1,2-DCE	0.3296	0.3459	0.3690	0.3945	0.4116	0.3701	0.0337	9.1071	
TCE=	0.3019	0.3186	0.3376	0.3634	0.3597	0.3366	0.0269	7.9875	
1,2-DEP	0.4012	0.4436	0.4382	0.4860	0.4791	0.4496	0.0343	7.6269	
DCBN	0.4106	0.4574	0.4560	0.4778	0.4582	0.4520	0.0248	5.4901	
C-130CP=	0.4124	0.4707	0.4776	0.5016	0.5017	0.4728	0.0365	7.7281	
1-130CP=	0.4231	0.4828	0.5023	0.5217	0.5316	0.4923	0.0430	8.7296	
112TCE	0.3419	0.3910	0.4199	0.4370	0.4528	0.4085	0.0437	10.7026	
1122TCE=	0.3263	0.3105	0.3429	0.3581	0.3677	0.3411	0.0232	6.8051	
DBCN	0.5895	0.6226	0.6162	0.6135	0.6429	0.6149	0.0192	3.1087	
CL BENZ	0.8540	0.9375	0.9603	0.9576	0.9731	0.9365	0.0478	5.1094	
CHBR3	1.1082	1.1417	1.0116	0.9874	1.0331	1.0564	0.0657	6.2200	
1122TCE	0.5311	0.5889	0.5816	0.6304	0.6756	0.6015	0.0544	9.0436	
1,3-DCB	0.5591	0.5908	0.5824	0.6328	0.6545	0.6039	0.0388	6.4325	
1,4-DCB	0.4865	0.5194	0.5200	0.5735	0.5684	0.5336	0.0368	6.8916	
1,2-DCB	0.5607	0.5993	0.5814	0.6180	0.6394	0.5998	0.0307	5.1167	
<hr/>									
VINYL CL (P)	22.4190	19.3380	16.4396	15.0310	13.6234	17.3302	3.5801	20.6584	USE XRSD FROM HALL
11-DCE= (P)	6.2485	5.6412	5.4141	5.6040	4.7441	5.5304	0.5397	9.7590	USE XRSD FROM HALL
1-12-DCE= (P)	1.9721	1.6354	1.3922	1.5153	1.3518	1.5734	0.2491	15.8297	USE XRSD FROM HALL
TERT-BME (B)	8.9273	8.4619	7.4878	8.2940	7.8327	8.2007	0.5587	6.8127	
BENZENE	1.4616	1.3834	1.2601	1.3801	1.2315	1.3433	0.0954	7.0992	
TCE= (P)	2.7503	2.3661	2.0745	2.0775	1.7975	2.2132	0.3614	16.3280	USE XRSD FROM HALL
C-130CP= (P)	7.1444	7.2378	6.1037	6.0097	5.2743	6.3540	0.8296	13.0560	USE XRSD FROM HALL
TOLUENE	1.5664	1.4752	1.3449	1.2310	1.2793	1.3794	0.1391	10.0834	
1-130CP= (P)	4.5957	4.6218	4.0262	3.7450	3.4385	4.0854	0.5211	12.7540	USE XRSD FROM HALL
1122TCE= (P)	3.6725	3.1601	2.8053	2.8391	2.4247	2.9804	0.4465	15.6538	USE XRSD FROM HALL
CL BENZ	1.4782	1.4254	1.2662	1.3233	1.2482	1.3483	0.1003	7.4358	
ETHYL BENZ	1.7448	1.6448	1.5354	1.5966	1.4575	1.5959	0.1088	6.8179	
MP-XYLENE (B)	1.5696	1.4665	1.3623	1.5153	1.3457	1.4559	0.0915	6.2826	
O-XYLENE (B)	2.0050	1.9083	1.6618	1.7867	1.6572	1.8038	0.1528	8.4691	
1,3-DCB	1.7861	1.6419	1.4090	1.3877	1.2486	1.4947	0.2156	14.6249	
1,4-DCB	1.7472	1.6267	1.4723	1.5215	1.3516	1.5439	0.1507	9.7587	
1,2-DCB	2.1828	2.0104	1.7676	1.7450	1.5982	1.8608	0.2330	12.5204	

(P) DETECTED ON PID; QUANTITATION FROM HALL DETECTOR (B) INCLUDED FOR BTX; NOT ON 602/8020 LIST

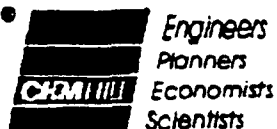


VOA INITIAL CALIBRATION FORM
 GC/MS-DB-1 MEGABORE CAPILLARY
 STANDARD: CS
 FILE NAME: ZIC0514A
 DATE: 05/14/91

COMPOUND NAME	RF 5 PPS	RF 20 PPS	RF 40 PPS	RF 100 PPS	RF 200 PPS	AVERAGE RF	STD DEV (N-1)	% RSD	XRSD MUST BE <30 USE AV RF IF XRSD >20
CH3CL	3.8237	3.7693	3.6873	2.8363	3.1957	3.4625	0.4296	12.4073	
VINYL CL	1.4331	1.4140	1.2932	1.4950	1.5355	1.4361	0.0925	6.4513	
CH3BR	2.2524	1.9590	1.8788	2.0297	2.0488	2.0338	0.1393	6.8314	
CL ETHANE	0.9444	0.9972	1.0058	1.1340	1.1964	1.0555	0.1051	9.9613	
TCFM	1.0521	1.0666	0.9994	1.1200	1.1172	1.0711	0.0501	4.6766	
1,1-DCE	0.5267	0.5241	0.5091	0.5747	0.5872	0.5444	0.0343	6.3094	
DCM	0.5789	0.6478	0.5538	0.5945	0.5791	0.5908	0.0350	5.9285	
T-12-DCE	0.3523	0.3783	0.3376	0.3917	0.4122	0.3744	0.0300	8.0013	
1,1-DCE	0.3287	0.3619	0.3434	0.3765	0.3807	0.3582	0.0220	6.1489	
CHCL3	0.2428	0.2686	0.2633	0.2855	0.3013	0.2723	0.0222	8.1695	
1,2-DCE	0.4072	0.4194	0.4001	0.4302	0.4444	0.4263	0.0252	5.9456	
1,1,1-TCE	0.3513	0.3802	0.3518	0.3948	0.4226	0.3801	0.0302	7.9529	
CCL4	0.2971	0.3167	0.2869	0.3146	0.3338	0.3098	0.0182	5.8903	
1,2-DCP	0.4051	0.4169	0.3805	0.4048	0.4317	0.4078	0.0188	4.6117	
TCE=DCM	0.3550	0.3514	0.3387	0.3640	0.4415	0.3701	0.0409	11.0577	
C-13DCP	0.4952	0.4497	0.4195	0.4436	0.4366	0.4489	0.0282	6.2829	
T-13DCP	0.5330	0.4665	0.4398	0.4427	0.4538	0.4671	0.0383	8.1926	
112TCE	0.3162	0.3311	0.3178	0.3438	0.3232	0.3264	0.0114	3.4778	
DBCM	0.6113	0.5670	0.5345	0.5436	0.6333	0.5784	0.0426	7.3420	
1122TCE	0.2666	0.2904	0.2792	0.3028	0.2960	0.2870	0.0143	4.9823	
CL BENZ	0.8371	0.7946	0.7471	0.7848	0.8925	0.8112	0.0556	6.8519	
CHBR3	0.9711	0.8225	0.7938	0.8210	0.8381	0.8493	0.0699	8.2330	
1122TCE	0.5403	0.5900	0.5683	0.6097	0.6085	0.5834	0.0294	5.0311	
1,3-DCB	0.5504	0.5517	0.5197	0.5448	0.5424	0.5418	0.0130	2.3933	
1,4-DCB	0.4977	0.5349	0.4800	0.5096	0.5336	0.5112	0.0236	4.6099	
1,2-DCB	0.5130	0.5349	0.5068	0.5415	0.5563	0.5305	0.0205	3.8588	
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VINYL CL (P)	42.8470	44.7183	32.9776	31.7006	28.8983	36.2284	7.0831	19.5514	USE XRSD FROM HALL
11-DCE (P)	7.2872	7.3121	5.5258	5.3963	5.2267	6.1496	1.0552	17.1591	USE XRSD FROM HALL
T-12-DCE (P)	2.2093	1.7602	1.3811	1.4859	1.4327	1.6539	0.3632	20.7545	USE XRSD FROM HALL
TERT-BME (B)	7.4182	5.3670	5.0852	5.2022	5.2971	5.6739	0.9808	17.2858	
BENZENE	1.7083	1.2831	1.2543	1.2954	1.3543	1.3791	0.1876	13.6032	
TCE (P)	2.6526	2.0899	1.7759	1.8261	1.8051	2.0299	0.3701	18.2333	USE XRSD FROM HALL
C-13DCP (P)	8.0174	7.2538	5.9302	5.6422	5.5525	6.4792	1.0985	16.9543	USE XRSD FROM HALL
T-13DCP (P)	4.4076	4.6503	3.4799	3.4442	3.4251	3.8814	0.5976	15.5971	USE XRSD FROM HALL
TOLUENE	1.7829	1.4427	1.2876	1.4048	1.3428	1.4522	0.1941	13.3467	
1122TCE (P)	4.2707	3.5975	2.7696	2.7334	2.7043	3.2151	0.6986	21.7274	USE XRSD FROM HALL
CL BENZ	1.6726	1.3609	1.2247	1.3113	1.2910	1.3721	0.1749	12.7493	
ETHYL BENZ	1.8581	1.6861	1.4472	1.5558	1.5034	1.6101	0.1644	10.2119	
MP-XYLENE (B)	1.6694	1.7752	1.6262	1.7225	1.7269	1.7040	0.0574	3.3702	
O-XYLENE (B)	1.9284	1.7642	1.5096	1.5767	1.6033	1.6765	0.1690	10.0836	
1,3-DCB	2.1066	1.7143	1.3696	1.4275	1.3930	1.6022	0.3144	19.6225	
1,4-DCB	1.7378	1.6328	1.4855	1.4697	1.3828	1.5417	0.1417	9.1931	
1,2-DCB	2.3876	1.9444	1.7784	1.7163	1.6331	1.8920	0.2997	15.8396	

(P) DETECTED ON PID; QUANTITATION FROM HALL DETECTOR

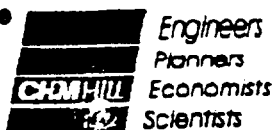
(B) INCLUDED FOR BTX; NOT ON 602/8020 LIST


 CONTINUING CALIBRATION FORM
 GC#1/DB-624 MEGABORE

 INIT CAL: 1/19/91
 DATE: 06/29/91
 FILE NAME: 1CC0629A

COMPOUND NAME	AVERAGE RF	RF 20 PPS	% D	METHOD &D LIMITS *	RRT MINS
CH3CL	2.3451	1.7459	25.8488	41	0.1490
VINYL CL	1.8463	1.3041	29.3646	32	0.1740
CH3BR	1.9980	1.3160	34.1375	42	0.2320
CL ETHANE	1.2115	0.9489	21.6777	23	0.2550
TCFM	0.8827	0.8180	7.3287	34	0.2940
1,1-DCE=	0.5469	0.6130	12.0836	37	0.3560
DCM	0.4514	0.3925	13.0539	23	0.4130
T-12-DCE=	0.3755	0.3695	1.5756	36	0.4390
1,1-DCE	0.4148	0.3822	7.8623	16	0.4770
CHCL3	0.3133	0.3206	2.3176	28	0.5610
1,1,1-TCE	0.4302	0.4494	4.4630	29	0.5710
CCL4	0.3510	0.3473	1.0566	32	0.5860
1,2-DCE	0.3701	0.4056	9.5775	29	0.6050
TCE=	0.3366	0.3204	4.8198	23	0.6600
1,2-DCP	0.4496	0.4723	5.0364	26	0.6760
DCBM	0.4520	0.4437	1.8391	24	0.7020
C-13DCP=	0.4728	0.5335	12.8407	36	0.7400
T-13DCP=	0.4923	0.5492	11.5498	36	0.7890
112TCE	0.4085	0.4121	0.8658	22	0.8030
1122TCE=	0.3411	0.3381	0.8697	30	0.8150
DBCM	0.6169	0.5868	4.8852	35	0.8360
CL BENZ	0.9365	0.9531	1.7734	28	0.8900
CHBR3	1.0564	0.8759	17.0828	27	0.9650
1122TCE	0.6015	0.5214	13.3177	51	1.0170
1,3-DCB	0.6039	0.5897	2.3555	28	1.1040
1,4-DCB	0.5336	0.5427	1.7037	31	1.1130
1,2-DCB	0.5998	0.5703	4.9061	32	1.1480
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VINYL CL	17.3302	24.3294	40.3875	ND	0.1730
1,1-DCE=	5.5304	7.0597	27.6524	ND	0.3550
T-12-DCE=	1.5734	1.6568	5.3034	ND	0.4380
TERT-BME	8.2007	4.4681	45.5154	50	0.4470
BENZENE	1.3433	1.1736	12.6386	23	0.6020
TCE=	2.2132	2.2379	1.1178	ND	0.6590
C-13DCP=	6.3540	7.0501	10.9557	ND	0.7400
TOLUENE	1.3794	1.3037	5.4820	23	0.7680
T-13DCP=	4.0854	4.7118	15.3308	ND	0.7890
1122TCE=	2.9804	3.0735	3.1243	ND	0.8150
CL BENZ	1.3483	1.3083	2.9616	20	0.8900
ETH BENZ	1.5959	1.4278	10.5305	37	0.9040
MP-XYLENE	1.4559	1.1779	19.0954	50	0.9160
O-XYLENE	1.8038	1.5579	13.6324	50	0.9510
1,3-DCB	1.4947	1.4935	0.0744	28	1.1040
1,4-DCB	1.5439	1.4165	8.2467	31	1.1140
1,2-DCB	1.8608	1.7892	3.8491	32	1.1490

* ND=NOT DETERMINED FOR 601 COMPOUNDS RESPONDING ON PID



CONTINUING CALIBRATION FORM
CC#2/DB-1 MEGABORE

INIT CAL: 05/25/91
DATE: 07/01/91
FILE NAME: 2CC0701A

COMPOUND NAME	AVERAGE RF	RF 20 PPB	% D	METHOD & D LIMITS *	RRT MINS
CH3CL	4.8118	4.2618	11.4307	41	0.1620
VINYL CL	1.8151	2.1078	16.1250	32	0.1910
CH3BR	2.4722	2.8291	14.4341	42	0.2340
CL ETHANE	1.3462	1.5067	11.9176	23	0.2540
TCFK	1.2251	1.3684	11.6986	34	0.3270
1,1-DCE=	0.6272	0.6065	3.2963	37	0.3750
DCM	0.5925	0.4588	22.5733	23	0.3900
T-12-DCE=	0.3814	0.4203	10.1981	36	0.4500
1,1-DCE	0.3787	0.4010	5.8661	16	0.4620
CHCL3	0.2920	0.3169	8.5004	25	0.5330
1,2-DCE	0.4602	0.4239	7.8928	29	0.5730
1,1,1-TCE	0.3926	0.4055	3.2841	29	0.5850
CCL4	0.3308	0.3480	5.2090	32	0.6190
1,2-DCP	0.4512	0.4743	5.1232	26	0.6600
TCE-/DCBM	0.3788	0.4100	8.2105	24	0.6730
C-13DCP=	0.4821	0.5200	7.8672	36	0.7260
T-13DCP=	0.5027	0.5389	7.1879	36	0.7590
112TCE	0.3675	0.3686	0.2991	22	0.7670
DBCM	0.6167	0.5951	3.5089	35	0.8070
1122TCE=	0.3202	0.3500	9.2838	30	0.8510
CL BENZ	0.8834	0.9468	7.1784	28	0.8950
CHBR3	0.8703	0.8758	0.6413	27	0.9370
1122TCE	0.6375	0.6001	5.8688	51	0.9680
1,3-DCB	0.6033	0.6313	4.6477	28	1.1120
1,4-DCB	0.5509	0.5911	7.2969	31	1.1180
1,2-DCB	0.5866	0.6294	7.3096	32	1.1470
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VINYL CL	40.0924	50.2581	25.3555	ND	0.1910
1,1-DCE=	5.9781	6.5389	9.3806	ND	0.3750
T-12-DCE=	1.5691	1.9527	24.4445	ND	0.4500
TERT-BME	5.9915	5.2470	12.4258	50	0.4690
BENZENE	1.3774	1.3662	0.8134	23	0.6110
TCE=	1.9170	2.4751	29.1170	ND	0.6740
C-13DCP=	6.3506	7.6223	20.0254	ND	0.7260
T-13DCP=	3.9243	4.6965	19.6785	ND	0.7580
TOLUENE	1.4692	1.4117	3.9120	23	0.7820
1122TCE=	3.0324	3.8000	25.3145	ND	0.8510
CL BENZ	1.3618	1.5754	15.6886	20	0.8960
ETH BENZ	1.6348	1.6252	0.5890	37	0.9220
MP-XYLENE	1.9509	1.5239	21.8886	50	0.9350
O-XYLENE	1.7535	1.5797	9.9111	50	0.9670
1,3-DCB	1.5673	1.9012	21.3089	28	1.1120
1,4-DCB	1.6811	1.6908	0.5781	31	1.1190
1,2-DCB	1.9877	2.2236	11.8691	32	1.1480

* ND=NOT DETERMINED FOR 601 COMPOUNDS RESPONDING ON PID

SOP No. 0- 0001
Rev. No. 3
Date: 07-02-91

ATTACHMENT B



Engineers
Planners
Economists
Scientists

VOA DETECTION LIMIT VERIFICATION FORM
GC1/D8-624 MEGABORE COLUMN

DATE 2/20/91

FILE NAME 1DL0220A

COMPOUND NAME	AMT SPPB-1	AMT SPPB-2	AMT SPPB-3	AMT SPPB-4	AMT SPPB-5	AMT SPPB-6	AMT SPPB-7	AVERAGE AMT	STD DEV (N-1)	IDL 3*SD
DCDFM								0.000	0.000	0.000
CH3CL	1.600	1.820	1.710	1.670	1.670	1.560	1.640	1.667	0.084	0.251
VINYL CL	1.590	1.490	1.630	1.530	1.680	1.600	1.630	1.593	0.064	0.193
CH3BR	1.540	1.490	1.370	1.530	1.620	1.550	1.580	1.554	0.041	0.124
CL ETNAME	2.200	2.110	2.260	2.170	2.280	2.170	2.370	2.223	0.087	0.260
TCFM	0.790	0.700	0.810	0.760	0.810	0.760	0.760	0.770	0.038	0.115
1,1-DCE=	0.750	0.690	0.810	0.700	0.770	0.700	0.720	0.734	0.044	0.133
DCM	0.690	0.730	0.830	0.690	0.780	0.650	0.710	0.726	0.061	0.183
T-12-DCE=	0.750	0.660	0.730	0.710	0.760	0.700	0.710	0.714	0.030	0.090
1,1-DCE	0.750	0.660	0.710	0.700	0.760	0.720	0.730	0.719	0.033	0.100
CHCL3	0.720	0.680	0.730	0.720	0.780	0.740	0.750	0.731	0.031	0.092
1,1,1-TCE	0.700	0.690	0.750	0.730	0.780	0.730	0.740	0.731	0.030	0.091
CCL4	0.670	0.670	0.730	0.720	0.770	0.730	0.740	0.719	0.037	0.110
1,2-DCE	0.720	0.680	0.750	0.740	0.810	0.760	0.760	0.746	0.040	0.120
TCE=	0.710	0.690	0.750	0.730	0.760	0.710	0.710	0.720	0.021	0.062
1,2-DCP	0.700	0.710	0.770	0.760	0.800	0.770	0.780	0.754	0.037	0.111
DCBM	0.670	0.690	0.760	0.750	0.790	0.760	0.770	0.761	0.044	0.132
C-130CP=	0.700	0.720	0.800	0.780	0.820	0.760	0.760	0.763	0.042	0.127
T-120CP=	0.750	0.720	0.820	0.800	0.820	0.780	0.800	0.784	0.037	0.112
112TCE	0.770	0.730	0.820	0.800	0.850	0.810	0.810	0.799	0.038	0.115
1122TCE=	0.770	0.750	0.860	0.790	0.850	0.800	0.780	0.800	0.041	0.122
DBCM	0.830	0.810	0.910	0.880	0.960	0.910	0.860	0.880	0.052	0.155
CL BENZ	0.840	0.800	0.870	0.840	0.910	0.860	0.840	0.851	0.034	0.102
CHBR3	0.920	0.820	0.880	0.870	0.970	0.970	0.920	0.907	0.055	0.164
1122TCE	0.900	0.830	0.840	0.800	0.840	0.870	0.790	0.839	0.038	0.114
1,3-DCB	0.870	0.810	0.860	0.810	0.830	0.820	0.810	0.830	0.025	0.075
1,4-DCB	0.900	0.780	0.870	0.820	0.840	0.830	0.780	0.831	0.044	0.132
1,2-DCB	0.920	0.800	0.890	0.820	0.880	0.840	0.790	0.849	0.049	0.147
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VINYL CL	1.900	1.760	1.650	1.710	1.880	1.690	1.760	1.761	0.095	0.284
11-DCE=	0.720	0.910	1.830	0.820	0.900	0.700	0.800	0.840	0.116	0.348
T-12-DCE=	0.950	0.900	0.890	0.880	0.910	0.840	0.870	0.891	0.034	0.103
TERT-BME	1.730	1.760	1.810	1.760	1.880	1.670	1.690	1.757	0.072	0.215
BENZENE	0.960	0.920	0.920	0.920	0.950	0.880	0.890	0.920	0.029	0.087
TCE=	0.890	0.870	0.880	0.860	0.880	0.830	0.840	0.867	0.020	0.059
C-130CP=	0.890	0.860	0.880	0.900	0.900	0.840	0.850	0.874	0.024	0.073
TOLUENE	0.930	0.890	1.080	1.060	1.080	0.960	0.930	0.990	0.081	0.242
T-130CP=	0.890	0.840	0.890	0.890	0.920	0.850	0.850	0.876	0.029	0.088
1122TCE=	0.880	0.860	0.910	0.890	0.930	0.830	0.840	0.877	0.036	0.109
CL BENZ	0.920	0.860	0.900	0.880	0.930	0.840	0.810	0.877	0.043	0.130
ETHYL BENZ	0.970	0.870	0.920	0.920	0.960	0.850	0.860	0.907	0.048	0.145
MP-XYLENE	1.850	1.740	1.770	1.780	1.830	1.710	1.650	1.761	0.069	0.207
O-XYLENE	1.050	0.830	0.870	0.850	0.920	0.810	0.830	0.880	0.083	0.249
1,3-DCB	0.940	0.830	0.870	0.830	0.850	0.790	0.810	0.846	0.049	0.147
1,4-DCB	0.930	0.850	0.930	0.890	0.940	0.890	0.880	0.901	0.033	0.099
1,2-DCB	0.980	0.860	0.880	0.900	0.920	0.830	0.820	0.884	0.055	0.166



Engineers
Planners
Economists
Scientists

VOA DETECTION LIMIT VERIFICATION FORM
DC2/DB-1 MEGABORE COLUMN

DATE 02/17/91
FILE NAME ZDL0217A

COMPOUND NAME	AMT 1PPB-1	AMT 1PPB-2	AMT 1PPB-3	AMT 1PPB-4	AMT 1PPB-5	AMT 1PPB-6	AMT 1PPB-7	AVERAGE AMT	STD DEV. (N-1)	IDL 3*SD
CH3CL	1.860	1.610	1.630	1.760	1.740	1.780	1.730	1.730	0.086	0.259
VINYL CL	1.920	1.570	1.750	1.720	1.770	1.860	1.860	1.779	0.116	0.349
CH3BR	2.120	1.650	1.800	1.900	2.070	2.040	2.060	1.946	0.170	0.511
CL ETHANE	3.030	2.570	2.750	2.900	3.180	3.120	3.180	2.961	0.233	0.700
TCPM	1.020	0.820	0.900	0.900	1.010	1.020	1.030	0.957	0.083	0.249
1,1-DCE=	0.980	0.820	0.920	0.960	1.020	1.040	1.120	0.980	0.095	0.286
DCM	0.930	0.800	0.840	0.930	0.930	0.960	1.050	0.920	0.081	0.244
1-12-DCE=	0.900	0.750	0.860	0.890	0.950	0.990	1.010	0.907	0.088	0.264
1,1-DCE	0.970	0.800	0.920	0.960	1.100	1.070	1.100	0.989	0.110	0.331
CHCL3	0.960	0.810	0.910	0.860	1.000	1.000	1.060	0.943	0.088	0.263
1,2-DCE	1.020	0.800	0.930	0.930	1.010	1.110	1.070	0.981	0.104	0.312
1,1,1-TCE	1.030	0.820	0.950	0.930	1.010	1.060	1.120	0.989	0.098	0.295
CCL4	0.990	0.820	0.930	0.990	1.010	1.070	1.130	0.991	0.099	0.297
1,2-DCP	1.010	0.900	0.960	0.950	1.120	1.150	1.130	1.031	0.101	0.303
TCE=DCBM	2.060	1.760	2.000	1.980	2.160	2.240	2.190	2.056	0.163	0.489
C-13DCP=	0.970	0.830	0.990	0.930	1.050	1.110	1.020	0.989	0.088	0.264
1-13-DCP=	1.040	0.830	1.030	0.990	1.150	1.220	1.090	1.050	0.124	0.373
112-TCE	1.010	0.870	1.000	0.960	1.070	1.090	1.080	1.011	0.079	0.236
DBCM	1.000	0.840	1.000	1.000	1.110	1.070	1.100	1.017	0.092	0.275
1122TCE=	1.060	0.930	1.060	0.990	1.150	1.130	1.200	1.074	0.094	0.282
CL BEN2	0.980	1.030	1.040	1.030	1.080	1.190	1.190	1.077	0.082	0.247
CHBR3	0.960	0.920	0.980	0.920	1.060	1.070	1.050	0.994	0.065	0.196
1122TCE	0.970	0.900	0.970	0.910	1.040	1.060	1.010	0.980	0.061	0.183
1,3-DCB	0.790	0.860	0.890	0.860	1.100	1.010	0.980	0.927	0.107	0.321
1,4-DCB	0.850	0.840	0.840	0.860	1.000	0.950	0.980	0.903	0.071	0.213
1,2-DCB	0.940	0.890	0.950	0.960	1.150	1.030	1.090	1.001	0.092	0.277

11-DCE=	0.980	0.930	0.990	0.900	0.950	1.040	0.827	0.368	1.103
1-12-DCE=	0.910	0.960	1.030	1.010	1.030	1.070	0.859	0.382	1.147
TERT-BME	1.950	1.710	1.870	1.980	2.000	1.970	2.210	1.956	0.450
BENZENE	1.030	0.890	0.950	1.000	1.000	1.010	1.090	0.996	0.063
TCE=	0.990	0.860	0.970	0.980	0.950	0.970	1.030	0.964	0.052
C-13DCP=	0.960	0.850	0.960	0.970	0.960	1.010	1.060	0.967	0.064
TOLUENE	1.050	0.870	0.950	0.980	1.040	1.070	1.130	1.013	0.086
1-13DCP=	0.970	0.870	0.950	0.970	0.970	1.000	1.010	0.963	0.046
1122TCE=	1.030	0.910	0.970	1.030	1.010	1.040	1.080	1.010	0.055
CL BENZ	0.980	0.870	0.940	0.990	0.970	1.030	1.100	0.983	0.072
ETHYL BENZ	1.010	0.900	0.970	1.020	1.000	1.060	1.060	1.003	0.056
MP-XYLENE	2.110	1.860	2.100	1.970	1.990	2.090	2.230	2.050	0.120
O-XYLENE	1.010	0.920	1.000	1.000	0.990	1.020	1.100	1.006	0.053
1,3-DCB	1.010	0.910	1.000	1.040	0.990	1.050	1.130	1.019	0.067
1,4-DCB	1.000	0.900	0.980	1.020	1.010	1.090	1.080	1.011	0.064
1,2-DCB	1.020	0.920	1.020	1.020	1.030	1.060	1.070	1.020	0.049

THE DETERMINATION OF POLYNUCLEAR AROMATIC HYDROCARBONS AND
SELECTED HETEROCYCLICS AT LOW PARTS-PER-TRILLION CONCENTRATIONS
USING GAS CHROMATOGRAPHY / MASS SPECTROMETRY

OCTOBER 1991

CH2M HILL, INC.
MONTGOMERY, ALABAMA 36116

THE DETERMINATION OF POLYNUCLEAR AROMATIC HYDROCARBONS AND SELECTED HETEROCYCLICS

1.0 Summary of the Method

This procedure describes the method used to analyze water samples using capillary gas chromatography/mass spectrometry to detect selected group of compounds at low part per trillion levels.

A measured volume of sample, 1.7 L, is serially extracted with methylene chloride at a pH greater than 10 using a separatory funnel. The methylene chloride extract is dried and concentrated to a volume of 1 mL. A 20 uL aliquot of the extract is removed for a sensitive GC/FID screen, to determine if dilution is required. After screening, an appropriate amount of internal standard solution is added to the extract. The extract is then concentrated to a final volume of 50 uL and analyzed by GC/MS. Qualitative identification of target compounds appearing in the extract is performed using relative retention time and mass spectral interpretation. Quantitation is performed by use of the multiple internal standard technique.

The working linear range of this method is defined by the calibration standards used in preparing the initial calibration curve. The highest concentration injected is 3.0 ug/ml.

2.0 Target Analytes and Method Detection Limits

Tables 1 and 2 list the target analytes and the nominal estimated detection limits for the analysis of water samples. The detection limits must be verified by performance of validation experiments. The validation process requires the analysis of a minimum of seven replicates of a known concentration of the compounds in water. The concentration of the replicate analyses must be one to five times the estimated minimum detection limits, which is calculated by multiplying the standard deviation of the replicate results by three. The reported nominal detection limits are considered valid if they are larger than the values determined by the replicate spiking experiment.

Method performance at concentrations well within the calibration range of the method must be determined to provide recovery ranges for quality assurance objectives. According to directions given in 40 CFR Part 136, at least four spikes into a representative matrix, must be performed to determine accuracy ranges for the different analytes.

3.0 Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, and glassware. A method blank must be prepared and analyzed with each extraction batch of samples to demonstrate there is no interference due to these materials.

3.1.1 Glassware must be scrupulously cleaned. In addition to normal cleaning procedures, all glassware must be washed with a Chromic Acid solution, then thoroughly rinsed with tap water, acetone, and finally methylene chloride, before use.

3.1.2 High purity solvents are required due to the low final volume, and must be demonstrated to be free of interfering contaminants prior to use.

3.2 Matrix interferences may be co-extracted from the sample. These must be taken into consideration when evaluating the GC/FID screening information, as the sample extract will be concentrated to a small final volume.

4.0 Apparatus and Materials

4.1 Samples should be collected in the field in accordance with the quality assurance project plan. Sample containers should be glass or similar inert material which will not offer interferences for the analysis. A 2-L amber glass bottle fitted with a screw cap lined with Teflon, is normally used.

4.2 Glassware

4.2.1 Separatory funnel, 2-L, with Teflon stopcock.

4.2.2 Pipets, 10-mL, and 1-mL for addition of base, and surrogate and spike fortifications.

4.2.3 Drying column, Chromatographic column, stopcock, 19mm ID, 250-mL reservoir or larger.

4.2.4 Evaporative flask, Kuderna-Danish, 500-mL size, ground glass joints.

4.2.5 Concentrator tube, Kuderna-Danish, 10-mL size, round glass joint.

4.2.6 Snyder column, Kuderna-Danish, three-ball macro, ground glass joint, floodless type.

- 4.2.7 Snyder column, Kuderna-Danish, two-ball micro, ground glass joint, floodless type.
- 4.2.8 Graduated cylinder, 1-L size.
- 4.2.9 Conical tubes, 5 mL graduated, ground glass joint with stopper.
- 4.2.10 Erlenmeyer flask, 250-mL size.
- 4.2.11 Centrifuge tubes, 40-mL size.
- 4.2.12 Silanized glass wool.
- 4.3 Boiling chips, 10/40 mesh, heat at 400 °C for 30 minutes before use to insure freedom from contamination.
- 4.4 Heated water bath, steam delivery should be controllable.
- 4.5 Analytical balance capable of weighing +/- 0.0001 gram.
- 4.6 Centrifuge, IEC HN-SII or equivalent.
- 4.7 pH paper, wide range
- 4.8 Teflon tape
- 4.9 GC/MS System
 - 4.9.1 Gas Chromatograph - An analytical system complete with temperature programmable gas chromatograph and all required accessories. The injection port must be designed for capillary split/splitless injection.
 - 4.9.2 Mass spectrometer - Capable of scanning from 35 to 450 amu every 7s or less and using a 70V (nominal) electron energy in the electron impact ionization mode.
 - 4.9.3 Data system - A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the chromatographic program. The computer must have software that allows searching any GC/MS data file for specific m/z and plotting such m/z abundances versus time or scan number.

5.0 Reagents

- 5.1 Reagent water which does not produce an interference for parameters of interest.

Sodium hydroxide solution (10 N) - Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 mL.

- 5.3 Methanol, methylene chloride, and acetone, pesticide quality(minimum).

- 5.4 Sodium sulfate, (ACS)granular, anhydrous. Purify by heating in a shallow tray for four hours at 400 °C.

6.0 Standards

- 6.1 Concentrated stock solutions may be prepared by accurately weighing the primary neat materials and dissolving in methylene chloride. Alternately, commercially prepared stock standards may be purchased if certified by the manufacturer.

- 6.2 Working Standard Solutions - Using the stock solutions, prepare secondary or working standard solutions by making appropriate dilutions in methylene chloride or methanol as appropriate.

- 6.2.1 Surrogate spike solution - The surrogate stock solution is prepared in the following manner: 0.2000 g of each neat compound is weighed, combined in a 100 ml volumetric flask, and diluted to the mark with methylene chloride. Final concentration = 2000 ug/ml. The stock solution is sealed in several 10 ml amber-colored ampules until needed.

An intermediate solution is prepared by adding 0.50 ml of the stock solution(2000 ug/ml) to a 100 ml volumetric flask, and diluting to the mark with methanol. Final concentration = 10 ug/ml.

The working solution is prepared by adding 1.0 ml of the intermediate solution(10 ug/ml) to a 100 ml volumetric flask and diluting to the mark with methanol. This surrogate spiking solution contains the following surrogate compounds at the given concentrations. (1.0 ml of this solution is added to all field and quality control samples)

<u>Compounds</u>	<u>ng/ml</u>
1- Fluoronaphthalene	100
d5-Nitrobenzene	100
2-Fluorobiphenyl	100
d14-Terphenyl	100

6.2.2 Matrix spiking solution - The matrix spiking solution is prepared in the following manner: 0.2000 g of each neat compound is weighed, combined in a 100 ml volumetric flask, and diluted to the mark with methylene chloride. Final concentration = 2000 ug/ml. The stock solution is sealed in several 10 ml amber-colored ampules until needed.

An intermediate solution is prepared by adding 5.0 ml of the stock solution (2000 ug/ml) to a 100 ml volumetric flask, and diluting to the mark with methanol. Final concentration = 100 ug/ml.

The working solution is prepared by adding 100 ul of the intermediate solution (100 ug/ml) to a 100 ml volumetric flask, and diluting to the mark with methanol. This matrix spiking solution contains the following compounds at the given concentrations.

<u>Compounds</u>	<u>ng/ml</u>
1H-Indene	100
Naphthalene	100
Quinoline	100
2-Methylnaphthalene	100
Acenaphthene	100
Phenanthrene	100
Carbazole	100
Pyrene	100
Benzo(a)anthracene	100
Chrysene	100
Benzo(b)fluoranthene	100
Benzo(e)pyrene	100
Benzo(a)pyrene	100
Indeno(1,2,3-cd)pyrene	100
Dibenzo(a,h)anthracene	100
Benzo(g,h,i)perylene	100

6.2.3 Internal standard stock solution - The internal standards used in this analysis are available from commercial vendors in a concentrated mixture. Ultra Scientific provides the internal standards at a

concentration of 4000 ug/ml in methylene chloride (Cat# US - 108). Intermediate and working solutions are prepared by mixing the following in 2.0 ml amber-colored vials.

400 ug/ml - 100 ul of the 4000 ug/ml Ultra stock solution is added to 900 ul of methylene chloride.

100 ug/ml - 250 ul of the 400 ug/ml intermediate solution is added to 750 ul of methylene chloride.

50 ug/ml - 500 ul of the 100 ug/ml working solution is added to 500 ul of methylene chloride.

10 ug/ml - 100 ul of the 100 ug/ml working solution is added to 900 ul of methylene chloride.

5 ug/ml - 100 ul of the 50 ug/ml working solution is added to 900 ul of methylene chloride

6.2.4 Calibration Standards - One of the concentrated stock solutions is purchased from Ultra Scientific (Cat# US - 106) containing the following compounds at a concentration of 2000 ug/ml:

Acenaphthene	Chrysene
Acenaphthylene	Fluoranthene
Anthracene	Fluorene
Benzo(a)anthracene	Naphthalene
Benzo(a)pyrene	Pyrene
Benzo(b)fluoranthene	Phenanthrene
Benzo(g,h,i)perylene	
Benzo(k)fluoranthene	
Dibenzo(a,h)anthracene	
Indeno(1,2,3-cd)pyrene	

The following stock solutions are prepared by weighing 0.0100 g of each neat compound, combining in a 5.0 ml volumetric flask, and diluting to the mark with methylene chloride. All mixes are at a concentration of 2000 ug/ml.

PNA MIX #1

Perylene
Triphenylene
Benzo(e)pyrene
3-Methyl cholanthrene

PNAMIX#2

Benzo(b)thiophene
1-Methylnaphthalene
Phenanthridine

PNA MIX #3

Benzofuran
Isoquinoline
Dibenzothiophene
7,12-Dimethylbenzo(a)anthracene

PNAMIX#4

2,3-Dihydro-1H-indene
Indole
Biphenyl
Carbazole

PNA MIX #5

1H-Indene
Quinoline
2-Methylnaphthalene

Dibenzofuran
Acridine

The first intermediate calibration solution is prepared by adding 0.5 ml of the following mixes to a 5.0 ml volumetric flask, and diluting to the mark with methylene chloride. Final concentration = 200 ug/ml.

PNA MIX #1
PNA MIX #2
PNA MIX #3
PNA MIX #4
PNA MIX #5

Ultra Scientific Mix
Surrogate Stock Mix
2000ug/ml (see 6.2.2)

A second intermediate calibration solution is prepared by adding 100 ul of the 200 ug/ml intermediate solution to 900 ul of methylene chloride. Final concentration = 20 ug/ml.

The working standard solutions are prepared by mixing the following in 2.0 ml amber-colored vials.

3.0
ug/ml - 150 ul of the 20 ug/ml intermediate is added to 850 ul of methylene chloride.

2.0 ug/ml -
200 ul of the 20 ug/ml intermediate is added to 1800 ul of methylene chloride.

* 1.0 ug/ml - 500 ul of the 2.0 ug/ml working solution is added to 500 ul of methylene chloride.

0.5 ug/ml - 250 ul of the 2.0 ug/ml working solution is added to 750 ul of methylene chloride.

0.1 ug/ml - 100 ul of the 1.0 ug/ml working solution is added to 900 ul of methylene chloride.

Internal Standard solution is added to achieve a concentration of 1.0 ug/ml in all working standard solutions.

* - The 1.0 ug/ml working standard is also to be used as the Continuing Calibration Standard.

6.2.5 Decafluorotriphenylphosphine(DFTPP) - Prepare a working solution of DFTPP in methylene chloride at a concentration of 50 ng/ul. A 1 ul(50 ng) aliquot is injected to validate GC/MS tuning.

7.0 Sample Extraction

- 7.1 A 2-liter separatory funnel is set up for each field sample, laboratory method blank, matrix spike, and matrix spike duplicate samples to be extracted. Transfer 1.7 liters of each sample into its funnel.
- 7.2 Check and record the pH of the sample, and then adjust to pH greater than 10 with the sodium hydroxide spiking solution.
- 7.3 Fortify each sample and the method blank with 1.0 ml of the surrogate spiking solution. Fortify any matrix samples with an additional 1.0 ml of the matrix spiking solution.
- 7.4 Add 60 mL of methylene chloride to the separatory funnel and shake vigorously for 3 minutes. Pressure will build up in the funnel rapidly, therefore invert the separatory funnel and vent frequently.
- 7.5 After shaking, allow the sample to separate into two phases. When phase separation has occurred, drain the methylene chloride layer into a 250 mL acid washed

- Erlenmeyer flask. If emulsions form, use 40 mL acid washed centrifuge tubes to collect the methylene chloride from the separatory funnel. Place the tubes into the centrifuge and spin for 2 minutes at 3,000 rpm. Remove the tubes and transfer the methylene chloride layer to the Erlenmeyer flask, and the water layer to the separatory funnel.
- 7.6 Repeat steps 7.4 and 7.5 two more times as described above.
 - 7.7 A drying column is prepared by plugging the end with silanized glass wool, and adding about 3 inches of muffled anhydrous sodium sulfate. Pre-rinse the packed column with approximately 50 mL of methylene chloride and discard.
 - 7.8 Assemble a Kuderna-Danish(K-D) concentrator by attaching a 10 mL concentrator tube to a 500 mL evaporative flask, and adding boiling chips. Place this under the drying column.
 - 7.9 Pour the combined extract through the drying column, collecting in the K-D concentrator. Rinse the 250-mL Erlenmeyer flask with two 20 mL portions of methylene chloride. After the extract has passed through the drying column, rinse with an additional 20 mL portion of methylene chloride.
 - 7.10 Attach a three-ball Snyder column to the evaporative flask, place the K-D on the water bath, and allow the extract to evaporate to approximately 1.0 mL. Remove the K-D and allow it to cool.
 - 7.11 Remove the Snyder column and rinse the flask and its lower joint with methylene chloride into the concentrator tube. Add one or two boiling chips, attach a two-ball micro Snyder column, and return to the hot water bath. Reconcentrate to an apparent volume of 0.5 mL and remove the concentrator tube and allow it to cool.
 - 7.12 Remove the Snyder column and rinse with 0.2 mL of methylene chloride. Quantitatively transfer the methylene chloride into an acid washed graduated conical tube. Rinse the concentrator tube with a second 0.2 mL aliquot of methylene chloride, and transfer. Establish a 1.0 mL final volume in the graduated conical tube, and seal around the glass stopper with Teflon tape prior to storing.

7.13 After the extraction has been completed, a 20 ul aliquot is removed from the 1.0 ml extract, for a sensitive GC/FID screen. Based on the results of the screening information, a portion of the appropriate internal standard solution is added to the 1.0 ml extract. The extract is then concentrated to it's final volume, typically 50 ul, just prior to analysis.

8.0 Instrument Operating Conditions

The following instrument conditions are provided as guidance for the analysis of the target compounds. Different conditions may be necessary for different brands or models of analytical instrumentation.

8.1 Gas Chromatograph - Capillary Column

8.1.1 Oven temperature program -

30°C (1.0 min) ---> 10°C/min --->
310°C (20min)

8.1.2 Injector temperature - 240°C

8.1.3 Injector - Grob splitless injection, split delay of 1.0 min.

8.1.4 Sample volume - 1 - 2 ul. (2 ul recommended)

8.1.5 Column - 30 Meter x 0.32mm ID bonded phase capillary column (Restec Rtx-5 or equivalent) 0.5-um film thickness.

8.1.6 Carrier flow - Helium @ 30 cc/sec

8.2 Mass Spectrometer

8.2.1 Electron Energy - 70 volts

8.2.2 Mass Range - 35 - 450

8.2.3 Scan Time - Scan time must be set to give at least 5 scans per chromatographic peak. A scan time of 0.5 seconds/scan for capillary columns is recommended.

9.0 Tuning and GC/MS Mass Calibration

It is necessary to establish that the GC/MS system is tuned and calibrated to meet the DFTPP standard mass spectral abundance criteria prior to any data acquisition of standards, blanks, or samples. The proper tuning and calibration must be verified for each 12 hours of instrument operation by analyzing 50 nanograms of DFTPP. The DFTPP ion abundance criteria is listed in Table 3.

10.0 Instrument Calibration

10.1 Prior to the analysis of samples and after the tuning criteria have been met, the linear calibration range of the instrument must be determined. The instrument must be calibrated using the same instrument conditions that are used to analyze the samples. Once the system has been initially calibrated, the calibration must be verified for each 12 hours of data acquisition.

10.2 Instrument calibration and sample analysis must be performed using multiple internal standards.

10.3 Initial calibration standards are prepared at a minimum of five concentration levels for each parameter analyzed. The concentrations of the standards for the initial calibration are 0.10, 0.50, 1.0, 2.0, and 3.0 ug/ml. The internal standards are at a concentration of 1.0 ug/ml in all solutions. Refer to section 6.2.5 for preparation details.

10.4 Analyze each calibration standard and tabulate the relative response factor(RRF), for each component at each level, using equation 2.

The relative response factor is calculated as follows.

Absolute Response Factor = $RF = \text{Amount/area}$
Eq. 1.

Relative Response Factor = $RRF = RF^{\text{target}}/RF^{\text{IS}}$
Eq. 2.

Note: Amount in equation 1 refers to the mass (e.g. ng) of compound mixed into the solution injected.

- 10.5 Calculate the standard deviation (S) and the relative standard deviation (%RSD) of RRFs for the compounds using the following equations:

$$S = \sqrt{\frac{\sum (RRF_i - RRF_m)^2}{N - 1}}^{1/2}$$

$$\text{and } \%RSD = \frac{S \times 100}{RRF_m}$$

Where:

RRF_i = Individual RRF

RRF_m = Mean RRF

N = Number of RRFs

$$\%RSD = \frac{S \times 100}{RRF_m}$$

- 10.6 Initial Calibration Criteria - The Relative standard deviation of each Calibration Check Compound (CCC) must be less than 30 percent. This criteria must be achieved for the calibration to be valid. Also, the mean RRF of the System Performance Check Compounds (SPCC) must meet minimum RRF criteria. The CCC and SPCC compounds with associated criteria are given below:

- 10.6.1 CCC Compounds - (RRF %RSD ≤ 30%)

Naphthalene
Acenaphthylene
Dibenzothiophene
Benzo(a)anthracene
Benzo(a)pyrene

- 10.6.2 SPCC Compounds - Must have Minimum Mean RRFs of 0.100.

Quinoline
Carbazole
3-Methyl Cholanthrene

The initial calibration is valid only after meeting both CCC and SPCC criteria. All initial calibration data and demonstration of achieving calibration criteria must be summarized and maintained in 3-ring binders for easy access and review.

11.0 Continuing Calibration

The working calibration curve or relative response factor for each analyte must be verified for each 12 hours of instrument operation by the analysis of a continuing calibration standard. The ongoing 12-hour continuing calibration standard must be compared to the initial calibration curve to verify that the operation of the measurement system is in control.

- 11.1 Analyze the 1.0 ug/ml continuing calibration standard and calculate the RRFs of each of the target analytes. All SPCC compounds must meet minimum relative response factor criteria. For each of the CCCs, calculate the percent difference of the continuing calibration RRF from the mean RRF from the initial calibration curve using the following equation:

$$\%D = \frac{[RRF_m - RRF]}{RRF_m} \times 100$$

Where:

RRF_m = The mean relative response factor from the initial calibration curve

RRF = The daily relative response factor

The percent difference for the CCC compounds must not exceed 25%.

The SPCC compounds must have a minimum RRF of 0.100.

Both SPCC and CCC criteria must be achieved every 12 hours for sample analysis to begin. If the criteria are not met, the system must be evaluated and corrective actions taken (possibly including acquisition of another initial calibration curve), before further sample analysis.

12.0 Quality Control

- 12.1 Every laboratory must operate a formal quality control program which meets or exceeds the requirements of the data quality objectives.
- 12.2 Instrument Blanks - Due to the low level of reporting limits, an instrument blank will be analyzed immediately after the continuing calibration standard, and prior to the analysis of any samples. The

instrument blank consists of the internal standards in methylene chloride, at a concentration of 1.0 ug/ml. The instrument blank is to verify the GC/MS system does not have any contamination from the daily standard. The instrument blank must not have any compound detected above the reporting limits.

- 12.3 Laboratory Method blank - The analytical system must be demonstrated to be free from contamination under the conditions of the extraction and analysis by the analysis of method blanks. An organic free water blank must be extracted with each batch of samples extracted on a given shift, not to exceed 20 samples per laboratory method blank.

Contamination by carry-over can also occur when high level and low level samples are analyzed in sequence. If target analytes are found in samples that are common to a previously analyzed high level sample, the samples must be reanalyzed and demonstration of a clean analytical system by the analysis of an instrument blank.

The results of blank analyses must be reported along with the results for samples. The blank results must not be subtracted from the sample analytical results.

A method blank must contain no greater than five times (5x) the stated reporting limits of commonly detected contaminants, such as Benzofuran, Naphthalene and Biphenyl. If the method blank exceeds these criteria, corrective actions must be taken and documented before further sample analysis.

- 12.4 Matrix Spikes/Matrix Spike Duplicates (MS/MSD) - At a minimum, one set of MS/MSD samples must be analyzed for each 20 samples to acquire data for measurement of accuracy and precision.

Calculate the Relative Percent Difference (RPD) for each of the designated MS/MSD compounds. An RPD value $\leq 25\%$ is considered to be acceptable. If the RPD value exceeds 25%, this must be discussed in the case narrative to the client.

The relative percent difference is calculated from the following equation:

$$RPD = \frac{[V1 - V2]}{[(V1 + V2)/2 - \text{sample}]} \times 100$$

The percent recoveries of the designated MS/MSD compounds are compared to the table of ranges below:

<u>Compound</u>	<u>Recovery</u>
1H-Indene	10-130
Naphthalene	10-130
Quinoline	10-130
2-Methylnaphthalene	10-130
Acenaphthene	10-130
Phenanthrene	10-130
Carbazole	10-130
Pyrene	10-130
Benzo(a)anthracene	10-130
Chrysene	10-130
Benzo(b)fluoranthene	10-130
Benzo(e)pyrene	10-130
Benzo(a)pyrene	10-130
Indeno(1,2,3-cd)pyrene	10-130
Dibenzo(a,h)anthracene	10-130
Benzo(g,h,i)perylene	10-130

* These limits are arbitrarily assigned, until sufficient spike data is available for an accurate statistical analysis.

If the percent recovery of a designated MS/MSD compound exceeds the criteria, this must be discussed in the case narrative to the client. The batch of samples do not have to be reanalyzed.

- 12.5 Surrogate Spike Results - All samples and blanks must be spiked with surrogate compounds prior to extraction. The surrogate recovery data must be summarized and statistically processed to determine control limits. If more than two surrogates exceed ± 3 standard deviations, the sample must be reanalyzed.

The current limits are listed below:

1-Fluoronaphthalene	4.0 - 64
2-Fluorobiphenyl	4.0 - 71
d14-Terphenyl	detected - 112
d5-Nitrobenzene	detected - 151

13.0 Qualitative Analysis

- 13.1 Qualitative identification is based on the following criteria.

- 13.1.1 The characteristic masses of each parameter must maximize within one scan of each other.
- 13.1.2 The retention time must fall within +/- 30 seconds of the retention time of the compound analyzed in the daily calibration standard.
- 13.1.3 The relative peak heights of the major characteristic masses must be with +/- 20% of the relative intensities of those in the reference mass spectrum. The reference mass spectrum is usually obtained from a reference library.

13.2 If a compound cannot be verified by all of the criteria in 13.1, but in the technical judgement of the mass spectral interpretation analyst, the identification is correct, then the compound will be reported.

14.0 Quantitative analysis

- 14.1 If the on-column concentration of any compound exceeds the initial calibration range, the sample must be diluted and reanalyzed. The dilution should be calculated such that the on-column concentration of compounds are kept in the upper half of the linear range. Additional internal standard must also be added to maintain a concentration of 1.0 ug/ml.
- 14.2 When a compound has been identified, the quantitation is based on the relative response factor that has been generated from the daily standard, as determined in section 9.3. Calculate the concentration in the sample using equation 4.

$$\text{PPT}_x \text{ sample} = \frac{(\text{ng}_{is}^{\text{sample}}) (\text{Area}_x^{\text{sample}})}{(\text{Area}_{is}^{\text{sample}}) (\text{RRF}_x) (\text{Volume extracted in L})} \text{---Eq. 4}$$

Where:

$\text{ng}_{is}^{\text{sample}}$ = amount of internal standard added to the extract.

$\text{Area}_x^{\text{sample}}$ = Area of the quantitation ion.

$\text{Area}_{is}^{\text{sample}}$ = Area of the quantitation ion the corresponding I.S.

RRF_x = Relative response factor from daily standard.

TABLE 1

Target Compounds	Quantitation Ions	
	Primary	Secondary
Benzofuran	118	89,63
2,3-Dihydro-1H-Indene	117	58,91
1H-Indene	116	58,89
Naphthalene	128	102,51
Benzo(b)thiophene	134	89
Quinoline	129	102,51
Isoquinoline	129	102,51
Indole	117	90,63
2-Methylnaphthalene	142	115,70
1-Methylnaphthalene	142	115,70
Biphenyl	154	76,51
Acenaphthylene	152	76,63
Acenaphthene	154	76,63
Dibenzofuran	168	139,84
Fluorene	166	82,139
Dibenzothiophene	184	139,82
Phenanthrene	178	89,152
Anthracene	178	89,152
Acridine	179	89,76
Phenanthridine	179	89,76
Carbazole	167	84,139
Fluoranthene	202	101,88
Pyrene	202	101,88
Benzo(a)anthracene	228	114,101
Chrysene/Triphenylene*	228	114,101
Benzo(b&k)fluoranthene	252	126,112
7,12-Dimethylbenzo(a)anthracene	256	119,126
Benzo(e)pyrene	252	126,112
Benzo(a)pyrene	252	126,112
Perylene	252	126,112
3-Methyl cholanthrene	268	126,252
Indeno(1,2,3-c,d)pyrene	276	138,125
Dibenzo(a,h)anthracene	278	139,125
Benzo(g,h,i)perylene	276	138,125

* Chrysene and Triphenylene coelute.

TABLE 2

Target Reporting Limits

Compound	ng/L
Benzofuran	4
2,3-Dihydro-1H-Indene	3
1H-Indene	3
Naphthalene	3
Benzo(b)thiophene	3
Quinoline	*
Isoquinoline	*
Indole	3
2-Methylnaphthalene	3
1-Methylnaphthalene	3
Biphenyl	3
Acenaphthylene	3
Acenaphthene	3
Dibenzofuran	3
Fluorene	3
Dibenzothiophene	3
Phenanthrene	3
Anthracene	3
Acridine	*
Phenanthridine	*
Carbazole	3
Fluoranthene	3
Pyrene	3
Benzo(a)anthracene	3
Chrysene/Triphenylene	6
Benzo(b&k)fluoranthene	6
7,12-Dimethylbenzo(a)anthracene	3
Benzo(e)pyrene	3
Benzo(a)pyrene	3
Perylene	3
3-Methyl cholanthrene	3
Indeno(1,2,3-c,d)pyrene	3
Dibenzo(a,h)anthracene	3
Benzo(g,h,i)perylene	3

* Method Detection Limit not determined at this time.

TABLE 3

DFTPP PERFORMANCE CRITERIA

Mass	Ion Abundance Criteria
51	30.0 - 60.0 percent of mass 198
68	less than 2.0 percent of mass 69
70	less than 2.0 percent of mass 69
127	40.0 - 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent relative abundance
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	greater than 1.0 percent of mass 198
441	present but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0 - 23.0 percent of mass 442

BOD
(Biochemical Oxygen Demand)

Method 405.1

Optimum Concentration Range: 10-300 mg/l
Sensitivity: 4 mg/l
Approximate Detection Limit: 10 mg/l
LIMS Test Code: BOD5
Holding Time: 2 Days to Begin Incubation

1.0 Method Summary:

The sample of waste, or an appropriate dilution, is incubated for 5 days at 20°C in the dark. The reduction in dissolved oxygen concentration during the incubation period yields a measure of the biochemical oxygen demand.

2.0 Bench Sheets:

Fill out the BOD bench sheet before beginning any analyses. Include all pertinent information such as sample size, dilution factors, dates of analysis, and sample ID. As the analysis proceeds, problems, variations, and other information are written on the bench sheet immediately. The analyst must initial and date the bench sheet when the sample run is set up and when the run is completed.

3.0 Spreadsheet:

All sample data and QC data should be entered into the BOD computer spreadsheet program within 24 hours after the analysis is completed. Calculations can be done manually or by use of the computer program. Date and initial the bench sheet when the data is entered into the spreadsheet program. When all QC data have been

entered, all calculations have been made, and the spreadsheet information has been saved to disk, the analyst will print hard copies of the related control charts, and other pertinent areas of the spreadsheet. These hard copies will be initialled, and clipped to the original bench sheet. When bench sheets are completed, the analyst will make copies for each client/sample group represented in the analytical run. The original bench sheet is put into the parameter binder with other pertinent information, for data review and for data entry. Copies are filed with client- or sample-specific files, to facilitate the final review of the final report for a client or sample group.

The analyst then reviews the data according to section 6 in the SOP manual. This review should be done within 24 hours of the analysis. When the analyst has completed the review, the data packet is placed in the parameter binder in the laboratory with the time noted on the bench sheet.

4.0 Data Review Process:

After the data review process has been completed (see Section 6 of the SOP Manual), within 24 hours, it is the responsibility of the analyst to enter the data into LIMS or to have the data-entry clerk enter the data into LIMS. The person who enters the data will initial and date the bench sheet, with a time, and the binder will be returned to the laboratory.

5.0 Quality Control Samples:

For BOD analyses, the following control samples are included on the bench sheet and should be run with each batch of samples:

- * method blank (dilution water blank)
- * QC check sample
- * duplicate samples

Acceptance limits for these quality control samples are as follows:

- * method blank (dilution water blank) - An unseeded dilution water blank should be used as a rough check on the quality of this water and the cleanliness of the incubation bottles. The DO uptake should not exceed 0.2 mg/l.
- * QC check sample - The spreadsheet has an area for entering data from the QC check sample. True value is given and the % recovery is calculated. This is charted on a control chart and statistical information is generated. The recovery on the QC sample must be within $\pm 3S$ for acceptance. When the QC recovery is outside this range, the system must be checked, a new QC sample made up, and the associated batch of samples must be re-analyzed. This must be documented on a corrective action report.
- * duplicate samples - Generally an RPD of 20 is considered the outside limit. The spreadsheet has an area for entry of duplicate analysis data. This will be charted after each analytical run. Acceptance limits are RPD inside $+ 3S$.

6.0 Calibration of DO meter:

- 6.1. Calibration against the Winkler method must be performed daily, and must be done prior to each set of DO measurements; results of calibration must be logged into the DO calibration log maintained close to the instrument.

6.1.1 Calibration is done on the dilution water. Initial calibration range should be between 7 mg/L and 9 mg/L. If the value is too low, dilution water should be aerated; if too high, a new water source should be obtained.

6.1.2 Taking meter reading:

- a. Fill 2 BOD bottles with dilution water. Be careful not to agitate or aerate the water as the bottles are filled. Fill from the bottom to the top using a glass tube or rod.
- b. Cap both bottles immediately.
- c. Insert probe into Bottle 1; turn agitator to ON; allow to agitate for 3 minutes.
- d. Read meter in mg/L. Record reading in DO logbook.
- e. On Bottle 2, perform the modified Winkler. See Attachment A for this procedure.
- f. With probe in Bottle 1, adjust the meter reading to agree with the results in mg/L O₂ obtained from the Winkler procedure.
- g. Do not turn off the DO meter between the calibration and other readings to be made on the same day.

6.2. Reagents:

- a) manganous sulfate solution: Dissolve 480 g manganous sulfate ($\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$) in distilled water and dilute to 1 L.
- b) Alkaline iodide-azide solution: Dissolve 500 g of NaOH, or 700 g of KOH and 135 g NaI, or 150 g of KI in distilled water and dilute to 1 L. To this solution, add 10 g of NaN_3 (sodium azide) dissolved in 40 mL distilled water.
- c) concentrated sulfuric acid
- d) starch solution: available commercially.
- e) potassium fluoride solution: Dissolve 40 g $\text{KF} \cdot 2\text{H}_2\text{O}$ in distilled water and dilute to 100 mL.
- f) sodium thiosulfate, stock solution, 0.75 N: Dissolve 186.15 g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in boiled and cooled distilled water and dilute to 1 L. Preserve by adding 5 mL chloroform.
- g) sodium thiosulfate standard titrant, 0.0375N: Prepare by diluting 50.0 mL of stock solution to 1 L. Preserve by adding 5 mL chloroform. Standard sodium thiosulfate, exactly 0.0375N is equivalent to 0.300 mg of DO per 1.00 mL. Standardize with 0.0375 N potassium biiodate.
- h) potassium biiodate, 0.0375 N: For stock solution dissolve 4.873 g of potassium biiodate, previously dried 2 hours at 103°C in 1000 mL distilled water. To prepare working standard, dilute 250 mL to 1000 mL for 0.0375 N.

6.3 Standardization of sodium thiosulfate:

- 6.3.1 Dissolve approximately 2 g KI in 100 to 150 mL distilled water; add 10 mL of 10% H_2SO_4 followed by 20 mL standard potassium biiodate. Place in dark for 5 minutes, dilute to 300 mL, and titrate with the standard sodium thiosulfate to a pale straw color. Add 1-2 mL starch solution and continue the titration drop by drop until the blue color disappears.
- 6.3.2 Run in duplicate. Duplicate determinations should agree within 0.05 mL.
- 6.3.3 To the calibration sample, add 2 mL manganous sulfate, followed by 2 mL of the alkaline iodide-azide solution, well below the surface of the liquid. Stopper with care to exclude air bubbles; mix well by inverting the bottle several times. When the precipitate settles, leaving a clear supernatant, shake again. When settling has produced at least 200 mL clear supernatant, carefully remove the stopper, and immediately add 2 mL concentrated H_2SO_4 , allowing the acid to run down the neck of the bottle, re-stopper, and mix by gentle inversion until the iodine is uniformly distributed throughout the bottle. Complete the analysis within 45 minutes.
- 6.3.4 Transfer 203 mL of the contents to a wide-mouth flask. Titrate with 0.025 N sodium thiosulfate to a pale straw color. Add 1-2 mL of starch solution and continue to titrate to the first disappearance of the blue color.

6.3.5 Calculation:

- a. Each ml of 0.025 N sodium thiosulfate is equivalent to 1 mg DO.
- b. This procedure should be compared with the results using the DO meter. All results should be recorded in the DO calibration log kept close to the instrument.

7.0 BOD Analysis:

7.1 Apparatus:

- a. incubation bottles: 250mL-300mL with ground-glass stoppers.

Clean bottles with detergent, rinse thoroughly and drain before use.

Use a water-seal. Invert bottles in a water bath or add water to the flared mouth of the BOD bottle. Place a paper or plastic cup over the flared mouth to reduce evaporation of the seal during incubation.

- b. air incubator or water bath: thermostatically controlled at $20 \pm 1^\circ\text{C}$. All light must be excluded during incubation to prevent photosynthetic production of DO.

7.2 Reagents:

- a. phosphate buffer solution

Dissolve 8.5 g KH_2PO_4 , 21.75 g K_2HPO_4 , 33.4 g $\text{Na}_2\text{HPO}_4 \cdot 7\text{H}_2\text{O}$, and 1.7 g NH_4Cl in about 500 mL distilled water and dilute to

1 L. Check the pH using a pH meter which has been properly calibrated. The pH should be 7.2 without further adjustment.

Discard any BOD reagent which shows any sign of biological growth in the stock bottle.

b. magnesium sulfate solution

Dissolve 22.5 g $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in distilled water and dilute to 1 L.

c. calcium chloride solution

Dissolve 27.5 g CaCl_2 in distilled water and dilute to 1 L.

d. ferric chloride solution

Dissolve 0.25 g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in distilled water and dilute to 1 L.

e. acid and alkali solutions, 1N

H_2SO_4 - Add 28 mL concentrated sulfuric acid to about 500 mL distilled water. Dilute to 1 L and mix well.

NaOH - Add 40 g of NaOH to about 500 mL distilled water. Dilute to 1 L and mix well.

f. sodium sulfite solution, 0.025N

Dissolve 1.575 g Na_2SO_3 in 1 L distilled water. This solution is not stable; prepare daily.

g. nitrification inhibitor: Available commercially

h. glucose-glutamic acid solution

Dry reagent-grade glucose and reagent-grade glutamic acid at 103°C for 1 hour. Add 150 mg glucose and 150 mg glutamic acid to distilled water and dilute to 1 L. Prepare fresh immediately before use.

i. sodium thiosulfate, 0.025 N:

Dissolve 6.205 g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in distilled water. Dilute to 1 L. Standardize.

7.3 Procedure:

7.3.1 Preparation of dilution water: Place desired volume of water in a suitable bottle and add 1 mL each of the phosphate buffer on day of analysis, MgSO_4 , CaCl_2 , and FeCl_3 solutions per liter of water. Seed dilution water, if desired. Test dilution water and store so that water of assured quality is always on hand.

7.3.2 Dilution water check: If dilution water has not been stored for quality improvement, add sufficient seeding material to produce a DO uptake of 0.05 to 0.1 mg/L in 5 d at 20°C. Do not seed dilution water that has been stored for quality improvement. Incubate a BOD bottle full of dilution water for 5 d at 20°C. Determine initial and final DO. The DO uptake in 5 days should not be more than 0.2 mg/L, and preferably not more than 0.1 mg/L.

Before use, bring dilution water temperature to 20°C. Saturate with DO by

shaking in a partially filled bottle or by aerating with filtered air. Protect water quality by using clean glassware, tubing, and bottles.

- 7.3.3 Glucose-glutamic acid check: THIS CHECK MUST BE DONE AT LEAST ONCE A WEEK. IF THERE IS NO EPA QC CHECK SAMPLE AVAILABLE, THE SUGAR SAMPLE MUST BE RUN WITH EVERY BATCH OF BODs.

Use a mixture of 150 mg glucose/L and 150 mg glutamic acid/L as a standard "check" solution. Determine the 5-day 20°C BOD of a 2% dilution of the glucose-glutamic acid standard check solution.

If the BOD value is outside the range of 200 ± 37 mg/L, reject any BOD determinations made with the seed and dilution water and seek the cause of the problem.

- 7.3.4 Seeding: Some samples do not contain a sufficient microbial population. For such wastes, seed the dilution water by adding a population of microorganisms. The preferred seed is effluent from a biological treatment system processing the waste.

Determine BOD of the seeding material as for any other sample. This is the seed control. From the value of the seed control and a knowledge of the seeding material dilution determine seed DO uptake. To determine a sample DO uptake, subtract the seed DO uptake from the total DO uptake. The DO uptake of the seeded dilution water should be between 0.6 and 1.0 mg/L.

7.3.5 Sample pretreatment:

- a. Samples containing caustic alkalinity or acidity: neutralize samples to pH 6.5 to 7.5 with H_2SO_4 or NaOH. The sample should not be diluted by more than 0.5%. The pH of seeded dilution water should not be affected by the lowest sample dilution.
- b. Samples containing residual chlorine: If residual chlorine is present, dechlorinate and seed the dilution water. Destroy chlorine by adding Na_2SO_3 solution.
 - 1) Determine required volume of Na_2SO_3 on a 100-mL to 1000-mL portion of neutralized sample, by adding 10 mL of 1+1 acetic acid, 10 mL potassium iodide solution (10g/100mL), and titrating with 0.025N Na_2SO_3 to the starch-iodine endpoint.
 - 2) Add to the neutralized sample the volume of Na_2SO_3 solution determined by the above test, mix, and after 10 to 20 minutes, check sample for residual chlorine.
- c. Samples supersaturated with DO: Samples containing more than 9 mg/L DO at 20°C may be brought to saturation by bringing sample to about 20°C in a partially filled bottle while shaking or aerating with compressed air.
- d. Nitrification inhibition: If the 5-day CBOD is requested, add 3.33 mg 2-chloro-6 (trichloro methyl) pyridine to each

bottle before capping or add sufficient amounts to the dilution water to make a final concentration of 10mg/L. Note the use of nitrogen inhibition in reporting results as Carbonaceous BOD.

- 7.3.6 Dilution technique: Make several dilutions of prepared sample to obtain a DO uptake in the range of 2 mg/L after 5 d incubation. In the absence of prior knowledge, use:

0.0 - 1.0% for strong industrial wastes
1.0 - 5.0 % for raw and settled wastewater
5.0 - 25.0% for biologically treated effluent
25.0 - 100% for polluted river waters

Prepare dilutions in volumetric flasks or directly in BOD bottles. Record dilution factors directly onto bench sheets as samples are diluted and prepared. A minimum of 3 dilutions are set up for each sample.

- 7.3.7 Determination of initial DO: If the sample contains materials that react rapidly with DO, determine initial DO immediately after filling BOD bottle. If rapid initial DO uptake is insignificant, the time period between preparing dilution and measuring initial DO is not critical.

- 7.3.8 Dilution water blank: Use a dilution water blank as a rough check on the quality of dilution water and BOD bottle cleanliness. With each batch of samples, incubate a bottle of unseeded dilution water. Determine initial and final DO uptake. The

DO uptake should not be more than 0.2 mg/L and preferably not more than 0.1 mg/L. If greater than 0.2 mg/l, the cause will be investigated and annotation made on the case narratives for affected samples.

7.3.9 Seed blank: With each batch of samples, incubate a bottle of seeded dilution water. Determine initial and final DO uptake.

7.3.10 Filled BOD sample bottles are covered with inverted paper cups to reduce evaporation, and placed in the BOD incubator at 20°C for 5 days. Final BOD values are determined at the end of the incubation period.

7.3.11 Using the DO probe to determine DO values:

- a. The DO meter is usually left on. After the meter has been calibrated, the samples can be read. Switch is set to DO MEASURED.
- b. Probe is placed into a beaker of distilled water and rinsed well. This is repeated after each sample reading.
- c. Probe is placed into a BOD sample bottle and the stirrer turned on. The meter is allowed to stabilize for a minimum of 3 minutes. Reading is recorded on bench sheet.

7.4 Calculation:

The bench sheets used (see example on following page) make the calculation of the DO very simple. Each column is used to record a specific value:

Column 0	= mls. of sample into BOD bottle
1	= dilution factor: 300ml divided by the mls of sample used = DF
2	= BOD bottle number
3	= initial DO uptake reading
4	= final DO uptake reading
5	= DO depletion (found by subtracting the value in column 4 from the value in column 3)
6	= % depletion (found by taking the depletion from column 5 and dividing it by the initial DO uptake from column 3. This is then multiplied by 100)
7	= BOD value (found by multiplying the DO depletion from column 5 by the dilution factor in column 1)

8.0 Reporting:

- a. BODs are reported in mg/L O₂ of original sample.
- b. Values below 2 mg/L are reported as < 2 mg/L O₂.

CH2M HILL/MGM SOP (BOD)
Rev. 0 1/30/89 -

OXYGEN, DISSOLVED

Method 360.2 (Modified Winkler, Full-Bottle Technique)

STORET NO. 00300

1. Scope and Application

- 1.1 This method is applicable for use with most wastewaters and streams that contain nitrate nitrogen and not more than 1 mg/l of ferrous iron. Other reducing or oxidizing materials should be absent. If 1 ml of fluoride solution is added before acidifying the sample and there is no delay in titration, the method is also applicable in the presence of 100–200 mg/l ferric iron.
- 1.2 The Dissolved Oxygen (DO) Probe technique gives comparable results on all samples types.
- 1.3 The azide modification is not applicable under the following conditions: (a) samples containing sulfite, thiosulfate, polythionate, appreciable quantities of free chlorine or hypochlorite; (b) samples high in suspended solids; (c) samples containing organic substances which are readily oxidized in a highly alkaline solution, or which are oxidized by free iodine in an acid solution; (d) untreated domestic sewage; (e) biological flocs; and (f) where sample color interferes with endpoint detection. In instances where the azide modification is not applicable, the DO probe should be used.

2. Summary of Method

- 2.1 The sample is treated with manganous sulfate, potassium hydroxide, and potassium iodide (the latter two reagents combined in one solution) and finally sulfuric acid. The initial precipitate of manganous hydroxide, $Mn(OH)_2$, combines with the dissolved oxygen in the sample to form a brown precipitate, manganic hydroxide, $MnO(OH)_2$. Upon acidification, the manganic hydroxide forms manganic sulfate which acts as an oxidizing agent to release free iodine from the potassium iodide. The iodine, which is stoichiometrically equivalent to the dissolved oxygen in the sample is then titrated with sodium thiosulfate or phenylarsine oxide (PAO).

3. Interferences

- 3.1 There are a number of interferences to the dissolved oxygen test, including oxidizing and reducing agents, nitrate ion, ferrous iron, and organic matter.
- 3.2 Various modifications of the original Winkler procedure for dissolved oxygen have been developed to compensate for or eliminate interferences. The Alsterberg modification is commonly used to successfully eliminate the nitrite interference, the Rideal-Stewart modification is designed to eliminate ferrous iron interference, and the Theriault procedure is used to compensate for high concentration of organic materials.
- 3.3 Most of the common interferences in the Winkler procedure may be overcome by use of the dissolved oxygen probe.

Approved for NPDES

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4. Sample Handling and Preservation

4.1 Where possible, collect the sample in a 300 ml BOD incubation bottle. Special precautions are required to avoid entrainment or solution of atmospheric oxygen or loss of dissolved oxygen.

4.2 Where samples are collected from shallow depths (less than 5 feet), use of an APHA-type sampler is recommended. Use of a Kemmerer type sampler is recommended for samples collected from depths of greater than 5 feet.

4.3 When a Kemmerer sampler is used, the BOD sample bottle should be filled to overflowing. (overflow for approximately 10 seconds). Outlet tube of Kemmerer should be inserted to bottom of BOD bottle. Care must be taken to prevent turbulence and the formation of bubbles when filling bottle.

4.4 At time of sampling, the sample temperature should be recorded as precisely as required.

4.5 Do not delay the determination of dissolved oxygen in samples having an appreciable iodine demand or containing ferrous iron. If samples must be preserved either method (4.5.1) or (4.5.2) below, may be employed.

4.5.1 Add 2 ml of manganous sulfate solution (6.1) and then 2 ml of alkaline iodide-azide solution (6.2) to the sample contained in the BOD bottle. Both reagents must be added well below the surface of the liquid. Stopper the bottle immediately and mix the contents thoroughly. The sample should be stored at the temperature of the collection water, or water sealed and kept at a temperature of 10 to 20°C, in the dark. Complete the procedure by adding 2 ml H_2SO_4 (see 7.1) at time of analysis.

4.5.2 Add 0.7 ml of conc. H_2SO_4 (6.3) and 1 ml sodium azide solution (2 g NaN_3 in 100 ml distilled water) to sample in the BOD bottle. Store sample as in (4.5.1). Complete the procedure using 2 ml of manganous sulfate solution (6.1), 3 ml alkaline iodide-azide solution (6.2), and 2 ml of conc. H_2SO_4 (6.3) at time of analysis.

4.6 If either preservation technique is employed, complete the analysis within 4–8 hours after sampling.

5. Apparatus

5.1 Sample bottles-300 ml \pm 3 ml capacity BOD incubation bottles with tapered ground glass pointed stoppers and flared mouths.

5.2 Pipets-with elongated tips capable of delivering 2.0 ml \pm 0.10 ml of reagent.

6. Reagents

6.1 Manganous sulfate solution: Dissolve 480 g manganous sulfate ($MnSO_4 \cdot 4H_2O$) in distilled water and dilute to 1 liter.

6.1.1 Alternatively, use 400 g of $MnSO_4 \cdot 2H_2O$ or 364 g of $MnSO_4 \cdot H_2O$ per liter. When uncertainty exists regarding the water of crystallization, a solution of equivalent strength may be obtained by adjusting the specific gravity of the solution to 1.270 at 20°C.

6.2 Alkaline iodide-azide solution: Dissolve 500 g of sodium hydroxide (NaOH) or 700 g of potassium hydroxide (KOH) and 135 g of sodium iodide (NaI) or 150 g of potassium iodide (KI) in distilled water and dilute to 1 liter. To this solution add 10 g of solution azide (NaN_3) dissolved in 40 ml of distilled water.

- 6.3 Sulfuric acid: concentrated.
 - 6.4 Starch solution: Prepare an emulsion of 10 g soluble starch in a mortar or beaker with a small quantity of distilled water. Pour this emulsion into 1 liter of boiling water, allow to boil a few minutes, and let settle overnight. Use the clear supernate. This solution may be preserved by the addition of 5 ml per liter of chloroform and storage in a 10°C refrigerator.
 - 6.4.1 Dry, powdered starch indicators such as "thyodene" may be used in place of starch solution.
 - 6.5 Potassium fluoride solution: Dissolve 40 g $\text{KF} \cdot 2\text{H}_2\text{O}$ in distilled water and dilute to 100 ml.
 - 6.6 Sodium thiosulfate, stock solution, 0.75 N: Dissolve 186.15 g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in boiled and cooled distilled water and dilute to 1 liter. Preserve by adding 5 ml chloroform.
 - 6.7 Sodium thiosulfate standard titrant, 0.0375 N: Prepare by diluting 50.0 ml of stock solution to 1 liter. Preserve by adding 5 ml of chloroform. Standard sodium thiosulfate, exactly 0.0375 N is equivalent to 0.300 mg of DO per 1.00 ml. Standardize with 0.0375 N potassium biiodate.
 - 6.8 Potassium biiodate standard, 0.0375 N: For stock solution, dissolve 4.873 g of potassium biiodate, previously dried 2 hours at 103°C, in 1000 ml of distilled water. To prepare working standard, dilute 250 ml to 1000 ml for 0.0375 N biiodate solution.
 - 6.9 Standardization of 0.0375 N sodium thiosulfate: Dissolve approximately 2 g (± 1.0 g) KI in 100 to 150 ml distilled water; add 10 ml of 10% H_2SO_4 , followed by 20.0 ml standard potassium biiodate (6.8). Place in dark for 5 minutes, dilute to 300 ml, and titrate with the standard sodium thiosulfate (6.7) to a pale straw color. Add 1–2 ml starch solution and continue the titration drop by drop until the blue color disappears. Run in duplicate. Duplicate determinations should agree within ± 0.05 ml.
 - 6.10 As an alternative to the sodium thiosulfate, phenylarsine oxide (PAO) may be used. This is available, already standardized, from commercial sources.
7. Procedure
 - 7.1 To the sample collected in the BOD incubation bottle, add 2 ml of the manganous sulfate solution (6.1) followed by 2 ml of the alkaline iodide-azide solution (6.2), well below the surface of the liquid; stopper with care to exclude air bubbles, and mix well by inverting the bottle several times. When the precipitate settles, leaving a clear supernatant above the manganese hydroxide floc, shake again. When settling has produced at least 200 ml of clear supernatant, carefully remove the stopper and immediately add 2 ml of conc. H_2SO_4 (6.3) (sulfamic acid packets, 3 g may be substituted for H_2SO_4)⁽¹⁾ by allowing the acid to run down the neck of the bottle, re-stopper, and mix by gentle inversion until the iodine is uniformly distributed throughout the bottle. Complete the analysis within 45 minutes.
 - 7.2 Transfer the entire bottle contents by inversion into a 500 ml wide mouth flask and titrate with 0.0375 N thiosulfate solution (6.7) (0.0375 N phenylarsine oxide (PAO) may be substituted as titrant) to pale straw color. Add 1–2 ml of starch solution (6.4) or 0.1 g of powdered indicator and continue to titrate to the first disappearance of the blue color.

- 7.3 If ferric iron is present (100 to 200 mg/l), add 1.0 ml of KF (6.5) solution before acidification.
- 7.4 Occasionally, a dark brown or black precipitate persists in the bottle after acidification. This precipitate will dissolve if the solution is kept for a few minutes longer than usual or, if particularly persistent, a few more drops of H_2SO_4 will effect dissolution.

8. Calculation

- 8.1 Each ml of 0.0375N sodium thiosulfate (or PAO) titrant is equivalent to 1 mg DO when the entire bottle contents are titrated.
- 8.2 If the results are desired in milliliters of oxygen gas per liter at 0°C and 760 mm pressure multiply mg/l DO by 0.698.
- 8.3 To express the results as percent saturation at 760 mm atmospheric pressure, the solubility data in Table 422:1 (Whipple & Whipple, p 446-447, Standard Methods, 14th Edition) may be used. Equations for correcting the solubilities to barometric pressures other than mean sea level are given below the table.
- 8.4 The solubility of DO in distilled water at any barometric pressure, p (mm Hg), temperature, $T^\circ C$, and saturated vapor pressure, u (mm Hg), for the given T , may be calculated between the temperature of 0° and 30°C by:

$$ml/l DO = \frac{(P - u) \times 0.678}{35 + T}$$

and between 30° and 50°C by:

$$ml/l DO = \frac{(P - u) \times 0.827}{49 + T}$$

9. Precision and Accuracy

- 9.1 Exact data are unavailable on the precision and accuracy of this technique; however, reproducibility is approximately 0.2 mg/l of DO at the 7.5 mg/l level due to equipment tolerances and uncompensated displacement errors.

Bibliography

1. Kroner, R. C., Longbottom, J. E., Gorman, R.A., "A Comparison of Various Reagents Proposed for Use in the Winkler Procedure for Dissolved Oxygen", PHS Water Pollution Surveillance System Applications and Development, Report #12, Water Quality Section, Basic Data Branch, July 1964.
2. Annual Book of ASTM Standards, Part 31, "Water", Standard D1589-60, Method A, p 373 (1976).
3. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 443, method 422 B (1975).

CHEMICAL OXYGEN DEMAND

Method 410.4 (Colorimetric)

Linear Concentration Range: 20 - 900 mg/l

Approximate Detection Limit: 20 mg/l

LIMS Test Code: COD4

Holding Time: 28 days

1.0 Scope and Application

1.1 This method covers the determination of COD in surface waters, domestic and industrial wastes.

1.2 The applicable range is 20 to 900 mg/l.

2.0 Summary Method

2.1 Sample, blanks and standards in sealed tubes are heated in an over or block-digester in the presence of dichromate at 150° C. After two hours, the tubes are removed from the over or digester, cooled and measured spectrophotometrically at 600 nm.

3.0 Sample Handling and Preservation

3.1 Collect the samples in glass bottles if possible. Use of plastic containers is permissible if it is known that no organic contaminants are present in the containers.

3.2 Samples should be preserved with sulfuric acid to a pH < 2 and maintained at 4° C until analysis.

4.0 Interferences

4.1 Chlorides are quantitatively oxidized by dichromate and represent a positive interference. Mercuric sulfate is added to the digestion tubes to complex the chlorides.

5.0 Apparatus

5.1 Drying oven or block digesgor, 500° C

5.2 Corning culture tubes, 16 x 100 mm or 25 x 150 mm with Teflon lined screw cap

5.3 Spectrophotometer

5.4 Muffle furnace, 500° C

5.5 Commercially available twist micro EPA approved digestion tubes (available from BioScience Inc., 174-318 Standard COD Vials).

6.0 Reagents

6.1 Digestion solution: Add 10.2 K₂Cr₂O₇, 167 ml conc. H₂SO₄ and 33.3 g HgSO₄ to 500 ml of distilled water, cool and dilute to 1 liter.

6.2 Catalyst solution: Add 22 g Ag₂SO₄ to a 4.09kg bottle of conc. H₂SO₄. Stir until dissolved.

6.3 Sampler wash solution: Add 500 ml of conc H₂SO₄ to 500 ml of distilled water.

6.4 Stock potassium acid phthalate: Dissolve 0.850 g in 800 ml of distilled water and dilute to 1 liter. 1 ml = 1 mg COD. Potassium acid phthalate must be crushed and dried for 72 hours at 120° C and desiccated before weighing.

6.4.1 Prepare a series of standard solutions that cover the expected sample concentrations by diluting appropriate volumes of the stock standard.

Standards

ml of Stock KHP/100 ml	Conc of mg/l COD
2	20
5	50
10	100
25	250
40	400
60	600
90	900

7.0 Procedure

7.1 Wash all culture tubes and screw caps with 20% H₂SO₄ before their first use to prevent contamination. Trace contamination may be removed from the tubes by igniting them in a muffle oven at 500° C for 1 hour.

7.2 Lab prepared Vials.

7.2.1 Add 2.5 ml of sample to the 16 x 100 mm tubes.

7.2.2 Add 1.5 ml of digestion solution (6.1) and mix.

- 7.2.3 Add 3.5 ml of catalyst solution (6.2) carefully down the side of the culture tubes.
- 7.2.4 Cap tightly and shake to mix layers
- 7.2.5 Process standards and blanks exactly as the samples.
- 7.2.6 Place in over or block digester at 150° C for two hours.
- 7.2.7 Cool, and place standards in sampler in order of decreasing concentration. Completely filling sampler tray with unknown samples.
- 7.2.8 Measure color intensity on Spectrophotometer at 600 nm.
- 7.3 Commercially Available Micro Vials
 - 7.3.1 Add 2.5 ml of sample to prepared vials.
 - 7.3.2 Replace cap firmly and shake to mix layers.
 - 7.3.3 Place in oven or block digester at 150° C for two hours.
 - 7.3.4 Cool
 - 7.3.5 Place layer of digested sample or standards in a standard 10 mm Quarts cell*.
 - 7.3.6 Measure color intensity on spectrophotometer at 600 nm.

* Care should be taken not to get the mercuric sulfate in the UV cell.

- 8.0 Bench Sheets: Fill out the COD bench sheet before beginning any analyses. Include all pertinent information such as sample size, dilution factors, dates of analysis, and sample ID. As the analysis proceeds, problems, variations, and other information are written on the bench sheet immediately. The analyst must initial and date the bench sheet when the sample run is set up and when the run is completed.
- 9.0 Spreadsheet: All sample data and QC data should be entered into the COD computer spreadsheet program within 24 hours after the analysis is completed. Calculations can be done manually or by use of the computer program. Date and initial the bench sheet when the data is entered into the spreadsheet program. When all QC data have been entered, all calculations have been made, and the spreadsheet information has been saved to disk, the analyst will print hard copies of the related control charts, and other pertinent areas of the spreadsheet. These hard copies will be initialled, and clipped to the original bench sheet. When bench sheets are

completed, the analyst will make copies for each client/sample group represented in the analytical run. The original bench sheet is put into the parameter binder with other pertinent information, for data review and for data entry. Copies are filed with client-or sample-specific files, to facilitate the final review of the final report for a client or sample group.

The analyst then reviews the data according to section 6 in the SOP manual. This review should be done within 24 hours of the analysis. When the analyst has completed the review, the data package is placed in the parameter binder in the laboratory with the time noted on the bench sheet.

10.0 Data Review Process: After the data review process has been completed (see Section 6 of the SOP Manual), within 24 hours, it is the responsibility of the analyst to enter the data into LIMS or to have the data-entry clerk enter the data into LIMS. The person who enters the data will initial and date the bench sheet, with a time, and the binder will be returned to the laboratory.

11.0 Quality Control Samples: For COD analyses, the following control samples are included on the bench sheets and should be run with each batch of samples:

- method blank (water blank)
- QC check sample
- duplicate samples

Acceptance limits for these quality control samples are as follows:

- method blank (water blank) - must be digested and analyzed with each digestion batch and have a result of < 20 mg/l.
- QC check sample - The spreadsheet has an area for entering data from the QC check sample. True value is given and the % recovery is calculated. This is charted on a control chart and statistical information is generated. The recovery on the QC sample must be within $\pm 3S$ for acceptance. When the QC recovery is outside this range, the system must be checked, a new QC sample made up, and the associated batch of samples must be re-analyzed. This must be documented on a corrective action report. QC sample must be digested and analyzed with each digestion batch. QC sample must be an independent check sample such as EPA Demand PE or a Demand PE from Analytical Products Group, Inc.
- Duplicate samples - Generally an RPD of 20 is considered the outside limit. The spreadsheet has an area for entry of

duplicate analysis data. This will be charted after each analytical run. Acceptance limits are RPD inside $+ 3S$.

TOTAL SUSPENDED SOLIDS
(Residue, Non-Filterable)

Method 160.2 (Gravimetric, Dried at 103-105°C)

Optimum Concentration Range: 4mg/L - 20000 mg/L
Sensitivity:
Approximate Detection Limit: 4 mg/L
LIMS Test Code: TSS
Holding Time: 7 Days

1.0 Method Summary:

A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to a constant weight at 103-105°C. The filtrate from this method may be used for TDS (Residue, Filterable).

2.0 Bench Sheets:

Fill out the TOTAL SUSPENDED SOLIDS bench sheet before beginning any analyses. Include all pertinent information such as sample size, dilution factors, dates of analysis, and sample ID. As the analysis proceeds, problems, weights, variations, and other information are written on the bench sheet immediately. The analyst must initial and date the bench sheet when the sample run is set up and when the run is completed.

3.0 Spreadsheet:

All sample data and QC data should be entered into the TOTAL SUSPENDED SOLIDS computer spreadsheet program within 24 hours after the analysis is completed. Calculations can be done manually or by use of the computer program. Date and initial the bench sheet when the data is entered into the spreadsheet program.

When all QC data have been entered, all calculations have been made, and the spreadsheet information has been saved to disk, the analyst will print hard copies of the related control charts, and other pertinent areas of the spreadsheet. These hard copies will be initialed, and clipped to the original bench sheet. When bench sheets are completed, the analyst will make

copies for each client/sample group represented in the analytical run. The original bench sheet is put into the parameter binder with other pertinent information, for data review and for data entry. Copies are filed with client- or sample-specific files, to facilitate the final review of the final report for a client or sample group.

The analyst then reviews the data according to section 6 in the SOP manual. This review should be done within 24 hours of the analysis. When the analyst has completed the review, the data packet is placed in the parameter binder in the laboratory with the time noted on the bench sheet.

4.0 Data Review Process:

After the data review process has been completed (see Section 6 of the SOP Manual), within 24 hours, it is the responsibility of the analyst to enter the data into LIMS or to have the data-entry clerk enter the data into LIMS. The person who enters the data will initial and date the bench sheet, with a time, and the binder will be returned to the laboratory.

5.0 Quality Control Samples:

For TOTAL SUSPENDED SOLIDS analyses, the following control samples are included on the bench sheet and should be run with each batch of samples:

- * method blank
- * QC check sample
- * duplicate samples

Acceptance limits for these quality control samples are as follows:

- * method blank - if the analyte of interest is detected in the method blank, any sample in which the analyte is present at < 10X the level detected in the blank must be re-analyzed. The spreadsheet has a section for entering blank result data.

- * QC check sample - The spreadsheet has an area for entering data from the QC check sample. True value is given and the % recovery is calculated. This is charted on a control chart and statistical information is generated. The recovery on the QC sample must be within $\pm 3S$ for acceptance. When the QC recovery is outside this range, the system must be checked, a new QC sample made up, and the associated batch of samples must be re-analyzed. This must be documented on a corrective action report.
- * duplicate samples - Generally an RPD of 20 is considered the outside limit. The spreadsheet has an area for entry of duplicate analysis data. This will be charted after each analytical run. Acceptance limits are RPD inside $\pm 3S$.

6.0 Analytical Procedure:

6.1 Apparatus:

- a. Glass fiber filter disks, without organic binder, such as Millipore AP-40, Whatman 934-AH, Gelman type A/E, or equivalent, 11 cm.
- b. Filter support: filtering apparatus with reservoir and a coarse (40-60 microns) fritted disk as a filter support.
- c. Suction flask
- d. Drying oven, 103-105°C
- e. Desiccator
- f. Analytical balance capable of weighing to 0.1 mg

6.1 Procedure:

6.1.1 Preparation of glass fiber filter disk:

- 1) Place the glass fiber filter on the

membrane filter apparatus or insert into bottom of a suitable Gooch crucible with wrinkled surface up.

- 2) While vacuum is applied, wash the disk with three successive 20 mL volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through.
- 3) Remove filter from membrane filter apparatus or both crucible and filter if Gooch crucible is used. Dry in an oven at 103-105°C for one hour. Remove to desiccator and store until needed.
- 4) Repeat the drying cycle until a constant weight is obtained (weight loss less than 0.5 mg). Weigh immediately before use. After weighing, handle the filter or crucible/filter with forceps or tongs only.

6.1.2 Selection of sample volume:

- 1) For an 11 cm filter, filter 100 mL of sample. If weight of captured residue is less than 1.0 mg, the sample volume must be increased to provide at least 1.0 mg of residue. Sample size can be decreased for samples with high solid content.
- 2) If other filter diameters are used, start with a sample volume equal to 7 mL/sq.cm. of filter area and collect at least a weight of residue proportional to the 1.0 mg stated above.

6.2 Procedure:

- a. Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fritted support.
- b. Shake the sample vigorously and quantitatively transfer the predetermined sample volume to the filter using a graduated cylinder. Remove

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Rev. 0 1/30/89

all traces of water by continuing to apply vacuum after sample has passed through.

- c. With suction on, wash the graduated cylinder, filter, residue, and filter funnel wall with three portions of distilled water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after water has passed through.
- d. Carefully remove the filter from the filter support. Alternatively, remove crucible and filter from the crucible adapter. Dry at least 1 hour at 103-105°C. Cool in a desiccator and weigh.
- e. Repeat the drying cycle until a constant weight is obtained (weight loss less than 0.5 mg).

6.3 Calculation:

Calculate TSS (non-filterable residue) as follows:

$$\text{TSS mg/L} = \frac{(A - B) \times 1000}{C}$$

where: A = weight of filter + residue, mg
B = weight of filter in mg
C = ml of sample filtered

7.0 Reporting:

- a. TSS (non-filterable residue) is reported in units of mg/L.
- b. Values below 4 mg/L are reported as <4.

8.0 Notes:

- 8.1 The practical range of the determination is 4 mg/L to 20 000 mg/L.
- 8.2 The filtrate from this method may be used for TDS (filterable residue).

- 8.3 Residue, non-filterable (TSS) is defined as those solids which are retained by a glass fiber filter and dried to constant weight at 103-105°C.
- 8.4 Non-representative particulates such as leaves, sticks, fish, lumps of fecal material should be excluded from the sample if it is determined that their inclusion is not desired in the final result.
- 8.5 Preservation of the sample is not practical. Analysis should begin as soon as possible. Refrigeration or icing to 4°C to minimize microbiological decomposition of solids, is recommended.

CH2M HILL
MONTGOMERY LABORATORY
STANDARD OPERATING PROCEDURES
WET CHEMISTRY DEPARTMENT

OIL AND GREASE (IR & GRAV.)

Method EPA 413.1 & 413.2

Working Linear Range: IR: 0.049 mg/L - 4.108 mg/L
Reporting Limit Water: IR: 0.05 mg/L Gravimetric: 0.1 mg/L
Reporting Limit Soil: IR: 1.6 mg/Kg Gravimetric: 3.3 mg/Kg
Reporting Unit: Water mg/L Soil mg/Kg
Matrix: Water and soil
Holding Time: 28 days from collection to analysis

1.0 SCOPE AND APPLICATION:

1.1 Gravimetric

- 1.1.1 This method includes the measurement of fluorocarbon-113 extractable matter from surface and saline waters, industrial and domestic wastes, soils, and sludges. It is applicable to the determination of relatively non-volatile hydrocarbons, vegetable oils, animal fats, soaps, greases and related matter.
- 1.1.2 The method is not applicable to measurement of light hydrocarbons that volatilize at temperatures below 70°C. Petroleum fuels from gasoline through #2 fuel oils are completely or partially lost in the solvent removal operation.
- 1.2.3 Some crude oils and heavy fuel oils contain a significant percentage of residue-type materials that are not soluble in fluorocarbon-113. Accordingly, recoveries of these materials will be low.

1.2 Infrared

- 1.2.1 This method includes the measurement of fluorocarbon-113 extractable matter from surface and saline waters, industrial and domestic wastes. It is applicable to the determination of hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related matter.
- 1.2.2 The method is applicable to measurement of most light petroleum fuels, although loss of about half of any gasoline present during the extraction manipulations can be expected.
- 1.2.3 While this method can be used to obtain an estimate of the oil and grease that would be measured gravimetrically, in many cases the

estimate more accurately describes the parameter, as it will measure volatiles more effectively and is not susceptible to interferences such as extractable sulfur. It can be used with the Petroleum Hydrocarbon procedure to obtain an oil and grease value and a petroleum hydrocarbon value on the same sample.

2.2 INFRARED:

2.2.1 The sample is acidified to a low pH (<2) and extracted with fluoro-carbon-113. The oil and grease is determined by comparison of the infrared absorbance of the sample extract with standards.

2.2.2 The IR method produces results which are not identical to those of the gravimetric method because composition of most QC samples (i.e., EPA QC Check Samples) varies from that of the IR standard. The IR method is based on the absorbance characteristics of C-H bonding. Therefore, identical results between IR and gravimetric methods will only occur when the sample and standard have the same chemical composition.

3.0 INTERFERENCES:

3.1 Trichlorotrifluoroethane dissolves not only oil and grease, but also other organic substances.

3.2 Do not use any plastic containers or plastic tubing for collection or processing of these samples.

4.0 SAFETY PRECAUTIONS:

Exercise normal laboratory safety precautions when performing this method.

5.0 SAMPLE COLLECTION AND HANDLING:

5.1 Sample Size:

Water: minimum of 1 liter; additional containers with equal sample volumes are required for client-specific QC such as matrix spike or duplicate.

Soil: minimum of 100 g.

5.2 Container: Glass with Teflon lined lid. Care should be taken to avoid lid liners which have been attached with glues which are extractable into the sample and by the fluoro-carbon-113.

5.3 Preservation:

Water: H_2SO_4 or HCl to pH <2.

Note: H_2SO_4 is preferred as HCl produces fumes upon addition of

sample to container.

Soil: none required -

5.4 Collection and Handling:

5.4.1 Water.

A representative sample of 1 liter volume should be collected in a glass bottle. If analysis is to be delayed for more than a few hours, the sample is preserved by the addition of 5 ml HCl (6.1) at the time of collection and refrigeration at 4°C.

Because losses of grease will occur on sampling equipment, the collection of a composite sample is impractical. Individual portions collected at prescribed time intervals must be analyzed separately to obtain the average concentration over an extended period.

5.4.2 Soil, Sediment, Sludge.

A representative sample of at least 200 g should be collected in a glass bottle.

6.0 APPARATUS:

6.1 Extraction (Gravimetric and IR).

6.1.1 Water.

6.1.1.1 Separatory funnel, 2000mL, with Teflon stopcock.

6.1.1.2 Filter paper, Whatman #40, 11cm.

6.1.2 Soil, Sludge, Sediment.

6.1.2.1 Sonicator.

6.1.2.2 Top-loading balance.

6.2 Analysis.

6.2.1 Infrared.

6.2.1.1 Infrared spectrophotometer, scanning. Non-scanning instruments can also be used, but can be subject to positive interferences in complex chemical wastewaters.

6.2.1.2 Cells, 10 mm, 50 mm, and 100 mm path length, sodium chloride or infrared grade glass.

6.2.2 Gravimetric.

- 6.2.2.1 Vacuum pump, or other source of vacuum.
- 6.2.2.2 Flask, boiling, 125 ml (Corning No. 4100 or equivalent).
- 6.2.2.3 Distilling head, Claisen or equivalent.
- 6.2.2.4 Filter paper, Whatman No. 40, 11 cm.
- 6.2.2.5 Analytical balance capable of accuracy to 0.1 gm.

7.0 ROUTINE PREVENTIVE MAINTENANCE:

7.1 Gravimetric.

Balance	Calibrate Maintenance	Daily Annually
Ovens	Temperature	Daily

7.2 Infrared.

IR	Change desiccant	Every six months
	Electronics maintenance	Every six months
	Check cells for scratches, smudges, or cracks	Every use

8.0 REAGENTS AND CALIBRATION STANDARDS:

8.1 Gravimetric.

- 8.1.1 Hydrochloric acid, 1:1. Mix equal volumes of conc. HCl and distilled water.
- 8.1.2 Fluorocarbon-113, (1,1,2-trichloro-1,2,2-trifluoroethane), b.p. 48°C.
- 8.1.3 Sodium sulfate, anhydrous crystal.

8.2 Infrared.

- 8.2.1 Sulfuric acid, 1:1: Mix equal volumes of concentrated H₂SO₄ and distilled water.
- 8.2.2 Fluorocarbon-113, b.p. 48°C.
- 8.2.3 Sodium sulfate, anhydrous crystal.
- 8.2.4 Calibration mixtures:

8.2.4.1 Reference oil: Pipet 15.0 mL n-hexadecane, 15.0 mL isooctane, and 10 mL chlorobenzene into a 50 mL glass stoppered bottle. Maintain the integrity of the mixture by keeping stoppered except when withdrawing aliquots.

8.2.4.2 Reference oil has a specific gravity of 0.8216. Therefore, standards can be made up by the following table for a 50 mm cell:\

uL Reference Oil into 100 mL Freon in 100 mL flask	mg/L
5.00	4.108
2.50	2.054
0.50	0.4108
0.25	0.2054
0.06	0.0493

9.0 CALIBRATION PROCEDURES:

9.1 Gravimetric.

No calibration is required for the gravimetric method.

9.2 Infrared.

9.2.1 Select appropriate working standards and cell pathlength according to the following table of approximate working ranges:

Pathlength	Range
10 mm	0.25-0.82 mg/L
50 mm	0.049-4.108 mg/L
100 mm	0.025-2.054 mg/L

9.2.2 Scan standards and samples from 3200 cm to 2700 cm with Freon in the reference beam and record the results on absorbance paper.

9.2.3 The absorbance of sample and standards are measured by constructing a straight baseline over the range of the scan and measuring the absorbance of the peak maximum at about 2930 cm and subtracting the baseline absorbance at that point.

9.2.4 Use a calibration plot of absorbance vs. mg oil prepared from the standards to determine the mg oil in the sample solution. This can be done using the computer spreadsheet program.

10.0 SAMPLE PREPARATION:

10.1 Water.

- 10.1.1 Mark the sample bottle at the water meniscus for later determination of sample volume. If the sample was not acidified at time of collection, add 5 ml hydrochloric acid (6.1) to the sample bottle. After mixing the sample, check the pH by touching pH-sensitive paper to the cap to insure that the pH is 2 or lower. Add more acid if necessary.
- 10.1.2 Pour the sample into a separatory funnel.
- 10.1.3 Tare a boiling flask (pre-dried in an oven at 103°C and stored in a desiccator).
- 10.1.4 Add 30 ml fluorocarbon-113 (6.2) to the sample bottle and rotate the bottle to rinse the sides. Transfer the solvent into the separatory funnel. Extract by shaking vigorously for 2 minutes. Allow the layers to separate, and filter the solvent layer into the flask through a funnel containing solvent moistened filter paper.
- NOTE: An emulsion that fails to dissipate can be broken by pouring about 1 g sodium sulfate (6.3) into the filter paper cone and slowly draining the emulsion through the salt. Additional 1 g portions can be added to the cone as required.
- 10.1.5 Repeat (7.4) twice more, with additional portions of fresh solvent, combining all solvent in the boiling flask.
- 10.1.6 Rinse the tip of the separatory funnel, the filter paper, and then the funnel with a total of 1-20 ml solvent and collect the rinsings in the flask.

10.2 Soil.

- 10.2.1 Weigh out 30 g of sample. Add 0.45 mL conc. HCL; mix well. Add enough MgSO₄ and mix well enough to make the sample a free-flowing sand-like material.
- 10.2.2 Add 30 mL Freon and extract by sonication for 3 minutes. Decant and filter the Freon through sodium sulfate and a Whatman #40 filter paper into a 100 mL Erlenmeyer flask. Stopper.
- 10.2.3 Repeat the extraction/sonication step twice using additional 30 mL aliquots of Freon. Combine the extracts.
- 10.2.4 Rinse the filter with 5 mL Freon. Combine with extracts. Bring volume to the 100 mL mark with Freon.

11.0 SAMPLE ANALYSIS:

11.1 Gravimetric.

- 11.1.1 Connect the boiling flask to the distilling head and evaporate the solvent by immersing the lower half of the flask in water at 70°C. Collect the solvent for reuse. A solvent blank should accompany each set of samples.
- 11.1.2 When temperature in the distilling head reaches 50°C or the flask appears dry remove the distilling head. Sweep out the flask for 15 seconds with air to remove solvent vapor by inserting a glass tube connected to a vacuum source. Immediately remove the flask from the heat source and wipe the outside to remove excess moisture and fingerprints.
- 11.1.3 Cool the boiling flask in a desiccator for 30 minutes and weigh.
- 11.1.4 Calculation for waters gravimetric

$$\text{mg/L total oil and grease} = \frac{R - B}{V}$$

where:

- R = residue, gross weight of extraction flask minus the tare weight, in milligrams.
- B = blank determination, residue of equivalent volume of extraction solvent, in milligrams.
- V = volume of sample, determined by refilling sample bottle to calibration line and correcting for acid addition if necessary, in liters.

11.1.4 Calculation for soils gravimetric

$$\text{mg/Kg total oil and grease} = \frac{(R - B) \times 100}{\text{Kg} \times \% \text{ solids}}$$

where:

- R = residue, gross weight of extraction flask minus the tare weight, in milligrams.
- B = blank determination, residue of equivalent volume of extraction solvent, in milligrams.
- Kg = weight of sample in kilograms.

11.2 Infrared.

- 11.2.1 Dilute the extract to 100 mL.
- 11.2.2 Fill the IR cell with each sample read and record absorbance.
- 11.2.3 Calculation

$$\text{mg/L total oil and grease} = \frac{R \times D}{V}$$

where:

- R = oil in solution, determined from calibration plot, in milligrams
- D = extract dilution factor
- V = volume of sample, determined by refilling sample bottle to calibration line and correcting for acid addition if necessary, in liters.

13.0 QUALITY CONTROL REQUIREMENTS:

13.1 The following QC samples should be run for this method:

- * method blank
- * QC check sample
- * duplicate samples

NOTE: For water samples, this must be a field duplicate sample since the method requires the extraction of the entire sample for the analysis.

- * matrix spike

NOTE: For water samples, this must be done on a field collected duplicate aliquot of the sample.

The reference oil is spiked directly into the separatory funnel containing the sample. Spike with 2.5 uL of the reference oil. To determine the amount spiked, use the following:

$$\frac{2.054}{L} \quad \text{or} \quad \frac{2.054}{K}$$

where: L = liters of sample

$$K = \frac{(\text{Kg soil extracted}) \times (\% \text{ solids})}{100}$$

13.2 Acceptance limits for these quality control samples are as follows:

- 13.2.1 * method blank - if the analyte of interest is detected in the method blank, any sample in which the analyte is present at < 10X the level detected in the blank must be re-analyzed. The spreadsheet has a section for entering blank result data.
- 13.2.2 * QC check sample - True value is given and the % recovery is calculated. This is charted on a control chart and statistical information is generated. The recovery on the QC sample must be within $\pm 3S$ for

acceptance. When the QC recovery is outside this range, the system must be checked and a new QC sample made up. For most analyses, this would require that the associated batch of samples be re-analyzed. However, the O&G method requires the use of the entire water sample for analysis. Consequently, when QC check sample recovery is out of acceptance range, only soils can actually be re-run. For water samples, the data must be flagged, and the case narrative should include explanation of the problem and information about other QC data and other sample data. This must be documented on a corrective action report.

- 13.2.3 * duplicate samples - Generally an RPD of 20 is considered the outside limit. This will be charted after each analytical run. Acceptance limits are RPD inside + 3S.

14.0 METHOD VALIDATION:

Each analyst must make an initial, one-time demonstration of the ability to generate acceptable accuracy and precision with this method.

15.0 REFERENCES:

- 15.1 Standard Methods for the Examination of Water and Wastewater, 16th Edition, 1985, Methods 503A and 503B.
- 15.2 Methods for Chemical Analysis of Water and Wastes, PB84, March 1983, Methods 413.1 and 413.2.

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Rev. No.: 0
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Date initiated: _____
Written by: K. Starcher

PERCENT SOLIDS (MOISTURE)

Method EPA 160.3 (209F, SM. 16th ed.)
(modified for use in accordance with
USEPA CLP Statement of Work For Inorganics Analysis, ILM01.0)

Optimum Concentration Range:
Sensitivity:
Reporting Detection Limit: 0.1 %
Reporting Units: %
Significant Figures: 2-3
Holding Time: 7 Days

1.0 Method Summary

An aliquot of solid or sludge sample is placed in a dried, weighed evaporating dish, the weight of the sample determined. The sample is then dried at 103 - 105 °C, weighed, and the % solids is determined.

2.0 Bench Sheets

Benchsheets or laboratory notebooks are to be used to enter raw data. For this SOP the term benchsheet will be used to signify either a benchsheet or a notebook entry.

Include all pertinent information in column headings; such as sample number, dish number, dish weight, weight of sample + dish, weight of sample, weight of dish + sample after drying, weight of dried sample. Identify all QC samples, e.g. duplicates, blank. The analyst must initial and date the bench sheet when the sample run is completed.

Show the equation for calculation the final result. Show all calculated final results on the benchsheet.

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3.0 Spreadsheet

All sample data and QC data should be entered into the Solids computer spreadsheet within 24 hours after the analysis is completed. Enter all QC data in the appropriate areas of the spreadsheet. Date and initial the bench sheet when the data is entered into the spreadsheet program.

4.0 Data Review Process

Within 24 hours the data review process should be completed. It then becomes the responsibility of the analyst to enter the data onto the Work In Progress (WIP) sheets or LIMS System or to see that a data entry person does so. The person who enters the data will initial and date the bench sheet and file it.

5.0 Quality Control Samples

The following quality control samples are required for % Solids analysis:

5.1 Duplicate: one per every 10 samples

5.2 Blank: one per analytical batch

6.0 Sample Handling and Preservation

6.1 Samples are collected in clean glass or plastic containers.

6.2 Preserve samples by storage at 4 °C.

6.3 The holding time is 7 days.

7.0 Apparatus

7.1 Evaporating dishes (such as porcelain, 90 mm diameter, platinum, high silica glass)

7.2 Drying oven, for operation at 103 to 105 °C

7.3 Analytical balance, capable of weighing to 0.01 g

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- 7.4 Desiccator, provided with a desiccant containing a color indicator of moisture concentration

9.0 Procedure:

- 9.1 Dry evaporating dish in drying oven at 103 to 105 °C for 1 hour. Cool dish to room temperature in desiccator (about 1 hour). Note time in desiccator. Weigh dish using analytical balance. Store weighed dish in desiccator until ready for use.
- 9.2 Place a representative aliquot of a well mixed sample in the weighed evaporating dish (10 - 25 grams). Weigh dish and sample, using analytical balance.
- 9.3 Dry in drying oven at 103 to 105 °C.
- 9.4 Cool dish and sample in desiccator for the same length of time as in step 9.1.
- 9.5 Weigh dish and sample using analytical balance.

10.0 Calculation:

- 10.1 Calculate the % solids using the following equation:

$$\% \text{ Solids} = \frac{(A - B)}{C - B} \times 100$$

where:

A	=	weight (grams) of dried solids + dish
B	=	weight (grams) of dish
C	=	weight (grams) of wet sample + dish

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10.2 Calculate % moisture using one of the following equations:

$$\% \text{ Moisture} = 100 - \% \text{ Solids}$$

or

$$\% \text{ Moisture} = \frac{(C - B) - (A - B)}{(C - B)} \times 100$$

where:

A	=	weight (grams) of dried solids + dish
B	=	weight (grams) of dish
C	=	weight (grams) of wet sample + dish

**CH2M HILL
MONTGOMERY LABORATORY
STANDARD OPERATING PROCEDURES
FOR TCLP**

Method: Appendix II-Method 1311 Toxicity
Characteristic Leaching Procedure (TCLP)
40 CFR Vol. 55, No. 126 Friday, June 29, 1990
Rules and Regulations

1. SCOPE AND APPLICATION

1.0 Scope and Application

- 1.1 The TCLP is designed to determine the mobility of both organic and inorganic contaminants present in liquid, solid, and multiphasic wastes.

2. SUMMARY OF METHOD

2.0 Summary of Method (See Figure 1.)

- 2.1 For liquid wastes (i.e., those containing less than 0.5 percent dry solid material), the waste, after filtration through a 0.6- to 0.8-um glass fiber filter, is defined as the TCLP extract.
- 2.2 For wastes containing greater than or equal to 0.5 percent solids, the liquid, if any, is separated from the solid phase and stored for later analysis: the solid phase, if necessary, is reduced in particle size. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatile contaminants. Following extraction, the liquid extract is separated from the solid phase by filtration through 0.6 to 0.8-um glass fiber filter.
- 2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

- 3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS

- 4.1 Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm.

4.2 Extraction Vessel:

- 4.2.1 Zero-Headspace Extraction Vessel (ZHE). This device is for use only when the waste is being tested for the mobility of volatile constituents (i.e., those listed in Table 1). The ZHE (depicted in Figure 3) allows for liquid/solid separation within the device and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel (See Step 4.3.1.). The vessels shall have an internal volume of 500-600 ml and be equipped to accommodate a 90-110 mm filter. The devices contain VITON® O-rings that should be replaced frequently.

For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psi or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses, and the manufacturer should be contacted.

The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

The ZHE uses gas pressure to actuate the ZHE piston.

- 4.2.2 Bottle Extraction Vessel. When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel.

The extraction bottles may be constructed from various materials, depending on the contaminants to be analyzed and the nature of the waste (See Step 4.3.3.). It is

recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene, shall not be used if organics are to be investigated. Bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in Step 4.3.2 is used for initial liquid/solid separation and final extract filtration.

4.3 Filtration Devices: It is recommended that all filtration be performed in a hood.

4.3.1 Zero-Headspace Extractor Vessel (ZHE): When the waste is evaluated for volatiles, the zero-headspace extraction vessel described in Step 4.2.1 is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psi).

Note: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

4.3.2 Filter Holder: When the waste is evaluated for other than volatile compounds, any filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more. The type of filter holder used depends on the properties of the material to be filtered (See Step 4.3.3.). These devices shall have a minimum internal volume of 300 ml and be equipped to accommodate a minimum filter size of 47 mm (Filterholders having an internal capacity of 1.5 l or greater and equipped to accommodate a 142 mm diameter filter are recommended.). Vacuum filtration can only be used for wastes with low solids content (<10 percent) and for highly granular liquid containing wastes. All other types of wastes should be filtered using positive pressure filtration.

4.3.3 Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials that will not leach or absorb waste components. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high-density polypropylene polyethylene (HDPE), polypropylene, or polyvinyl chloride may be used only when evaluating the mobility of metals. Borosilicate glass bottles are recommended for use over other types of glass bottles, especially when inorganics are constituents of concern.

4.4 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8 um or equivalent. Pre-filters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses

with deionized distilled water (A minimum of 1 l per rinse is recommended.). Glass fiber filters are fragile and should be handled with care.

- 4.5 pH meters: The meter should be accurate to ± 0.05 units at 25°C.
- 4.6 ZHE extract collection devices: TEDLAR® bags or glass, stainless steel or PTFE gas-tight syringes are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device.
- 4.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of nonaqueous liquid (i.e., <1 percent of total waste), the TEDLAR® bag or a 600 ml syringe should be used to collect and combine the initial liquid and solid extract.
- 4.6.2 If a waste contains a significant amount of nonaqueous liquid in the initial liquid phase (i.e., >1 percent of total waste), the syringe or the TEDLAR® bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysis should use one or the other, not both.
- 4.6.3 If the waste contains no initial liquid phase (is 100 percent solid) or has no significant solid phase (is 100 percent liquid), either the TEDLAR® bag or the syringe may be used. If the syringe is used, discard the first 5 ml of liquid expressed from the device. The remaining aliquots are used for analysis.
- 4.7 ZHE extraction fluid transfer devices. Any device capable of transferring that extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or peristaltic pump, a gas tight syringe, pressure filtration unit (See Step 4.3.2.), or other ZHE device).
- 4.8 Laboratory balance: Any laboratory balance accurate to within ± 0.01 grams may be used (All weight measurements are to be within ± 0.1 grams.).
- 5.0 REAGENTS
- 5.1 Reagent water. Reagent water is defined as water in which an interferant is not observed at or above the methods detection limit of the analyte(s) of interest. For nonvolatile extractions, ASTM Type II water or equivalent meets the definition of reagent water. Reagent water should be monitored periodically for impurities.
- 5.1.1 A water purification system (Continental® or equivalent will be with activated carbon.) may also be used to generate reagent water for volatile extractions.

- 5.2 Hydrochloric acid (1N), HCl, made from ACS reagent grade. 83 ml concentrated. Hydrochloric acid in distilled water dilute to 1 liter.
- 5.3 Nitric acid (1N), HNO₃, made from ACS reagent grade. 64 ml concentrated Nitric Acid in distilled water dilute to 1 liter.
- 5.4 Sodium hydroxide (1N), NaOH, made from ACS reagent grade. 40.0g NaOH in distilled water dilute to 1 liter.
- 5.5 Glacial acetic acid, HOAc, ACS reagent grade.
- 5.6 Extraction fluid.
- 5.6.1 Extraction fluid #1: Add 5.7 ml glacial HOAc to 500 ml of the appropriate water (See Step 5.1.), add 64.3 ml of 1N NaOH, and dilute to a volume of 1 liter or add 17.1 ml glacial HOAc to 500 ml of the appropriate water (see Step 5.1), add 7.716 g NaOH, mix and dilute to a volume of 3 liters. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05 .
- 5.6.2 Extraction fluid #2: Dilute 5.7 ml glacial HOAc with ASTM Type II water (See Step 5.1.) to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05 .

Notes: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

- 5.7 Analytical standards prepared according to the appropriate analytical method.
- 6.0 **SAMPLE HANDLING AND PRESERVATION**
- 6.1 Preservatives shall not be added to samples.
- 6.2 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.
- 6.3 When the waste is to be evaluated for volatile contaminants, care shall be taken to minimize the loss of volatiles. Samples shall be taken and stored in a manner to prevent the loss of volatile contaminants (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 4°C, until ready to be opened prior to extraction).

- 6.4 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic contaminant determinations must be acidified with nitric acid to a pH <2, unless precipitation occurs (See Step 8.14 if precipitation occurs.). Extracts or portions of extracts for organic contaminant determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. See below acceptable sample and extract holding times.

SAMPLE MAXIMUM HOLDING TIMES [Days]				
	From: Field collection To: TCLP extraction	From: TCLP extraction To: Preparative extraction	From: Preparative extraction To: Determinative analysis	Total elapsed time
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360

7. PROCEDURE

7.0 PRELIMINARY EVALUATIONS

Perform preliminary TCLP evaluations on a minimum 100 gram aliquotor waste. This may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids; (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration; (3) determination of whether the solid portion of the waste requires particle size reduction; and (4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste.

- 7.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

- 7.1.1 If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e., is 100% solids) proceed to Step 7.3.
- 7.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device described in Step 4.3.2. and is outlined in Steps 7.1.3 through 7.1.9.
- 7.1.3 Pre-weigh the filter and the container that will receive the filtrate.
- 7.1.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.
- 7.1.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight.
- 7.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.
- 7.1.7 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

Note: If waste material (> 1 percent of original sample weight has obviously adhered to the container used to transfer the sample of the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 7.1.5 to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval; proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2-minute period), stop the filtration.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

- 7.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

Note: Some wastes, such as oily wastes and some point wastes, will obviously contain some material that appears to be liquid. Even after applying vacuum or pressure filtration, as outlined in Step 7.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

- 7.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (See Step 7.1.3.) from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the wastes sample by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Step 7.1.5 or 7.1.7.

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid (Step 7.1.9)}}{\text{Total weight of waste (Step 7.1.5 or 7.1.7)}} \times 100$$

- 7.2 If the percent solids determined in Step 7.1.9 is equal or greater than 0.5%, then proceed either to Step 7.3 to determine whether the solid material requires particle size reduction or to Step 7.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids determined in Step 7.1.9 is less than 0.5%, then proceed to Step 8.9. If the nonvolatile TCLP is to be performed and to section 9.0 with a fresh portion of the waste if the volatile TCLP is to be performed.

- 7.2.1 Remove the solid phase and filter from filtration apparatus, and dry the solid phase and filter at $100 \pm 20^\circ\text{C}$ until two successive weighing yield the same value within ± 1 percent. Record the weight.

Note: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

- 7.2.3 Calculate the percent dry solids as follows:

$$\text{Percent solids} = \frac{(\text{Weight of dry waste + filter}) - \text{tared weight of filter}}{\text{Initial weight of waste (Step 7.1.5 or 7.1.7)}} \times 100$$

7.2.4 If the percent dry solids is less than 0.5 percent, then proceed to Step 8.9 if the nonvolatile TCLP is to be performed, and to Section 9.0 if the volatile TCLP is to be performed. If the percent dry solids is greater than or equal to 0.5% and if the nonvolatile TCLP is to be performed, return to the beginning of this Section (7.0) and, with a fresh portion of waste, determine the appropriate extraction fluid (Step 7.4). If only the volatile TCLP is to be performed, see the note in Step 7.4.

7.3 Determination of whether the waste requires particle size reduction (Particle size is reduced during this step.): Using the solid portion of the waste, evaluate the solid for particle size. Particle size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm² or is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle-size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken, see Step 9.8.

Note: Surface-area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required nor is it recommended. For materials that do not obviously meet the criteria, sample-specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

7.4 Determination of appropriate extraction fluid: If the solid content of the waste is greater than or equal to 0.5 percent and if TCLP extraction for nonvolatile constituents will take place (Section 8.0), perform the determination of the appropriate fluid (Step 5.6) to use for the nonvolatiles extraction as follows:

Note: TCLP extraction for volatile constituents uses only extraction fluid #1 (Step 5.6.1). Therefore, if TCLP extraction for nonvolatiles is not required, proceed to Section 9.0.

7.4.1 Weigh out a small subsample of the sample phase of the waste, reduce the solid (if necessary) to a particle-size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to 500-ml beaker or Erlenmeyer flask.

7.4.2 Add 96.5 ml of reagent water (ASTM Type II) to the beaker, cover either a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use the extraction fluid = 1. Proceed to Section 8.0.

7.4.3 If the pH from Step 7.4.2 is >5.0, add 3.5 ml 1N HCL slurry briefly, cover with a watchglass, heat to 50°C, and hold at 50°C for 10 minutes.

- 7.4.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0 , use extraction fluid #1. If the pH is >5.0 , use extraction fluid #2. Proceed to section 8.0.
- 7.5 If the aliquot of the waste used for the preliminary evaluation (Steps 7.1-7.4) was determined to be 100% solid at Step 7.1.1, then it can be used for the Section 8.0 extraction (assuming at least 100 grams remain) and the Section 9.0 extraction (assuming at least 25 grams remain). If the aliquot was subjected to the procedure in Step 7.1.7, then another aliquot shall be used for the volatile extraction procedure in Section 9.0. The aliquot of the waste subjected to the procedure in Step 7.1.7 might be appropriate for use for the Section 8.0 extraction if an adequate amount of solid (as determined by Step 7.1.9) was obtained. The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to Step 8.10 of the nonvolatile TCLP extraction.

8.0 PROCEDURE WHEN VOLATILES ARE NOT INVOLVED

A minimum sample size of 100 grams (solid and liquid phases) is required. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample (See Step 7.1 for determination of percent solids.); whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid; and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

- 8.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100 percent solid, see Step 7.1), weigh out a subsample of the waste (100 gram minimum) and proceed to Step 8.9.
- 8.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Step 4.3.2 and is outlined in Steps 8.3 to 8.8.
- 8.3 Pre-weigh the container that will receive the filtrate.
- 8.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (See Step 4.4.).

Note: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.

- 8.5 Weigh out a subsample of waste (100 gram minimum) and record the weight. If the waste contains <0.5 percent dry solids (Step 7.2), the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the TCLP extract. For wastes containing >0.5 percent dry solids (Steps 7.1 or 7.2), use the percent solids information obtained in Step 7.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.
- 8.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the waste is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.
- 8.7 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder (See Step 4.3.2.). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

Note: If waste material (>1 percent of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Step 8.5 to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter or when the liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2-minute period), stop the filtration.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

- 8.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (See Step 8.12.) or stored at 4°C until time of analysis.

Note: Some waste, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Step 8.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

- 8.9 If the waste contains <0.5 percent dry solids (See Step 7.2.), proceed to Step 8.13. If the waste contains >0.5 percent dry solids (See Step 7.1 or 7.2.), and if particle-size reduction of the solid was needed in Step 7.3, proceed to Step 8.10. If the waste as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase and proceed to Step 8.11.

- 8.10 Prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to surface area or particle size as described in Step 7.3. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

Note: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon-coated sieve should be used to avoid contamination of the sample.

- 8.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Step 7.1)} \times \text{weight of waste filtered (Step 8.5 or 8.7)}}{100}$$

Slowly add this amount of appropriate extraction fluid (See Step 7.4.) to the extractor vessel. Close the extractor bottle tightly. (It is recommended that Teflon tape be used to ensure a tight seal.) Secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at $22 \pm 3^\circ\text{C}$ during the extraction period.

Note: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may

be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

8.12 Following the 18 + 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Step 8.7. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (See Step 4.4.) if evaluating the mobility of metals.

8.13 Prepare the TCLP extracts as follows:

8.13.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Step 8.12 is defined as the TCLP extract. Proceed to Step 8.14.

8.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Step 8.12 with the initial liquid phase of the waste obtained in Step 8.7. This combined liquid is defined as the TCLP extract. Proceed to Step 8.14.

8.13.3 If the initial liquid phase of the waste, as obtained from Step 8.7, is not or may not be compatible with the filtered liquid resulting from Step 8.12, do not combine these liquids. Analyze these liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in Step 8.14.

8.14 Following collection of the TCLP extract, the pH of the extract should be recorded. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4°C) until analyzed. The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic contaminants. If an analysis of the undigested extract shows that the concentration of any regulated metallic contaminant exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to + 0.5 percent), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final analyte concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (l).

C_1 = The concentration of the contaminant of concern in the first phase (mg/l).

V_2 = The volume of the second phase (l).

C_2 = The concentration of the contaminant of concern in the second phase (mg/l).

- 8.15 Compare the contaminant concentration in the TCLP extract with the thresholds identified in the appropriate regulations. Refer to Section 10.0 for quality assurance requirements.

9.0 PROCEDURE WHEN VOLATILES ARE INVOLVED.

Use the ZHE device to obtain TCLP extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of nonvolatiles analytes (e.g., metals, pesticides, etc.).

The ZHE device has approximately a 500-ml interval capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (defined as that fraction of a sample from which no additional liquid may be forced out by an applied pressure of 50 psi), due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4°C) to minimize loss of volatiles.

- 9.1 Pre-weigh the (evacuated) filtrate collection container (see Step 4.6) and set aside. If using a TEDLAR® bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis. The containers listed in Step 4.6 are recommended for use under the conditions stated in 4.6.1-4.6.3.
- 9.2 Place the ZHE piston within the body of the ZHE (It may be helpful first to moisten the piston O-rings slightly with extraction fluid.). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Section 9.0, Step 7.1 and/or 7.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the

glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

- 9.3 If the waste is 100 percent solid (see Step 7.1), weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to Step 9.5.
- 9.4 If the waste contains <0.5 percent dry solids (Step 7.2), the liquid portion of waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing >0.5 percent dry solids (Step 7.1 and/or 7.2), use the percent solids information obtained in Step 7.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:
- 9.4.1 For wastes containing <0.5 percent solids (see Step 7.1), weigh out a 500-gram subsample of waste and record the weight.
- 9.4.2 For wastes containing >0.5 percent solids (see Step 7.1), determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE} = \frac{25}{\text{Percent solids (Step 7.1)}} \times 100$$

Weigh out a subsample of the waste of the appropriate size and record the weight.

- 9.5 If particle size reduction of the solid portion of the waste was required in Step 7.3, proceed to Step 9.6. If particle size reduction was not required in Step 7.3, proceed to Step 9.7.
- 9.6 Prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in Step 7.3.1. Wastes and appropriate reduction equipment should be refrigerated, if possible, to 4° C prior to particle size reduction. The means used to effect particle size must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

Note: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

When the surface area or particle size has been appropriately altered, proceed to Step 9.7.

- 9.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.
- 9.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet on the bottom). Do not attach the extract collection device to the top plate.

Note: If waste material (<1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Step 9.4 to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the wastes is 100 percent solid (See Step 7.1.), slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to Step 9.12.

- 9.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi, if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2-minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

- 9.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

Note: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case the material within the filtration devices is defined as a solid and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5 percent dry solids (See Step 7.2.), this filtrate is defined as the TCLP extract and is analyzed immediately (See Steps 9.13 through 9.15.) or stored at 4°C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #1 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (step 7.1)} \times \text{weight of waste filtered (Step 9.4 or 9.8)}}{100}$$

- 9.12 The following steps detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid =1 is used in all cases (See Step 5.6.).
- 9.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.
- 9.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 to 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psi (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into the hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.
- 9.12.3 Place the ZHE in the rotary agitation apparatus (if it is not already there) and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction occurs) shall be maintained at $22 \pm 3^\circ\text{C}$ during agitation.

- 9.13 Following the 18 ± 2 hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Check the ZHE for leaking as specified in Step 4.2.1 and perform the extraction again with a new sample of wastes. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases; if the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR® bag) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases or if there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Step 9.9. All extract shall be filtered and collected if the TEDLAR® bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase (See Steps 4.6 and 9.1.).

Note: All in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

- 9.14 If the original waste contained no initial liquid phase, the filtered liquid material obtained from step 9.13 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from Step 9.13 and the initial liquid phase (Step 9.9) are collectively defined as the TCLP extract.
- 9.15 Following collection of the TCLP extract immediately prepare the extract for analysis and store with minimal headspace at 4°C until analyzed. Analyze the TCLP extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simply volume-weighted average.

$$\text{Final analyte concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phases (l).

C_1 = The concentration of the contaminant of concern in the first phase (mg/l).

V_2 = The volume of the second phase (l).

C_2 = The concentration of the contaminant of concern in the second phase (mg/l).

- 9.16 Compare the contaminant concentrations in the TCLP extract with the thresholds identified in the appropriate regulations. Refer to section 10.0 for quality assurance requirements.

10.0 QUALITY ASSURANCE REQUIREMENTS

10.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel.

10.2 A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data is being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. The bias determined from the matrix spike determination shall be used to correct the measured values. (See sections 10.2.4 and 10.2.5.)

10.2.1 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

10.2.2 In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be not less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

10.2.3 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration of the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance.

10.2.4 Matrix spike recoveries are calculated by the following formula:

$$\%R (\% \text{ Recovery}) = 100 (X_s - X_u)/K$$

where:

X_s = corrected value, and
 X_u = measured value of the unspiked sample
 K = known value of the spike in the sample

10.2.5 Measured values are corrected for analytical bias using the following formula:

$$X_c = 100 (X_u / \%R)$$

X_c = corrected value, and
 X_u = measured value of the unspiked sample

- 10.3 All quality control measures described in the appropriate analytical methods shall be followed.
- 10.4 Samples must undergo TCLP extraction within the appropriate holding times.
- 10.5 Maintain all data, including quality assurance data, and keep it available for reference or inspection.

ORGANIC CARBON, TOTAL

(Combustion, Soil/Sediment)

Working Linear Range: 0 - 5000 mg/Kg
Reporting Limit: 5 mg/Kg
Reporting Units: mg/Kg, dry weight
Matrix: Soil/Sediment
Holding Time: 28 days from date of collection

1.0 PRINCIPLE, SCOPE AND APPLICATION

- 1.1 The high temperature furnace system operates on the concept of sparging, oxidation, and infrared detection. Oxidation is performed in a quartz combustion tube in an oxygen atmosphere, at approximately 800°C.
- 1.2 The advantage of the high temperature combustion is the oxidation of almost every substance likely to occur in a water sample. This makes this technique amenable to determination of sludges, sediments, and soils.

2.0 METHOD SUMMARY

A boat inlet system delivers the sample to a high temperature furnace. The sample is combusted at 800°C in an oxygen atmosphere so that solids as well as liquids can be analyzed.

3.0 INTERFERENCES:

Carbonate and bicarbonate carbon represent an interference to the determination of total organic carbon. They must be removed or accounted for in the final calculation.

4.0 SAFETY PRECAUTIONS:

Exercise normal laboratory safety precautions when performing this method.

5.0 SAMPLE COLLECTION AND HANDLING:

- 5.1 Sample Size: minimum 10 g
- 5.2 Container: Plastic or glass
- 5.3 Preservation: cool to 4°C
- 5.4 Collection and Handling: Fill container and tightly close lid. Preserve by refrigeration at 4°C.

6.0 APPARATUS

- 6.1 Total organic carbon analyzer with high temperature combustion furnace adapted for use with a boat inlet system for delivering sludge or soil/sediment samples (i.e. Dohrmann DC-80 TOC analyzer with a Sludge and Sediment Sampler accessory connected to a PRG-1 furnace module.)

7.0 ROUTINE PREVENTIVE MAINTENANCE:

Follow instrument manufacturer's instructions.

8.0 REAGENTS AND CALIBRATION STANDARDS:

- 8.1 Deionized water, organic free.
- 8.2 Organic Carbon Standards:
 - 8.2.1 2000 mg/L stock standard: Dry 3-5 g potassium hydrogen phthalate ($\text{KHC}_8\text{H}_4\text{O}_4$), (abbreviated KHP) to a constant weight. Carefully weigh 2.125 g dried KHP, and dissolve in deionized water. Add 0.5 mL concentrated phosphoric acid and dilute to 500 mL in a volumetric flask. Store in dark glass, under refrigeration. Replace monthly.

8.2.2 400 mg/L standard solution: Dilute 200 mL of 2000 mg/L stock standard solution to 1000 mL. Store in dark glass, under refrigeration. Prepare fresh weekly.

8.2.3 For other standard concentrations, dilute the 2000 mg/L stock solution appropriately.

8.3 5N Sulfuric Acid (H_2SO_4): Slowly add 140 mL conc. H_2SO_4 to approximately 800 mL deionized water. Mix. Cool and dilute to 1000 mL.

9.0 CALIBRATION PROCEDURE:

9.1 Follow instrument manufacturer's instructions for calibration.

9.2 Verify the calibration with an Initial Calibration Verification Standard (ICVS). The recommended acceptance criteria for the ICVS is 90-110% Recovery.

9.3 Analyze a Continuing Calibration Verification Standard (CCVS) after every 10 samples. The recommended acceptance criteria for the CCVS is 90-110% Recovery.

10.0 SAMPLE PREPARATION:

10.1 Add a 2-3 g aliquot of well mixed sample to a beaker.

10.2 Add 5N H_2SO_4 (8.3) dropwise until there is no more effervescing. Do not add too much acid at one time as this may cause loss of sample due to frothing. This acidification step will remove the carbonate and bicarbonate carbon from the sample.

10.3 Dry the sample at 70°C.

10.4 Mix the dried sample to obtain as much homogeneity as possible.

10.5 Proceed to Step 11.1.

10.6 Method Blank

10.6.1 Place approximately 10 g of sand in a casserole dish. Place in the muffle

furnace at 800 degrees C for 8 hours.
Cool in desicator.

- 10.6.2 Transfer approximately 0.5 g of the cooled sand to an aluminum weighing boat. Acidify the sample with 5N H_2SO_4 and dry at 70 degrees C.
- 10.6.3 Place the dried acidified sand in a platinum boat and weigh. Weight should be approximately 0.04 g.
- 10.6.4 Place the sample into the sample tube on the instrument. After the detector stabilizes, introduce the sample into the furnace. The detector will read the relative absorbance of CO_2 burned off by the furnace.

11.0 SAMPLE ANALYSIS

- 11.1 Set up TOC analyzer and furnace module according to instrument manufacturer's instructions. The furnace temperature should be 800°C.
- 11.2 Weigh a clean platinum boat containing quartz wool and record (or tare) this weight.
- 11.3 Add approximately 0.05 g of sample (from Step 10.5) to the boat. Weigh the boat and sample. Subtract the original weight of the boat from the weight of the boat plus sample to get the actual sample weight.
- 11.4 Place boat with sample into the inlet part of the furnace.
- 11.5 Wait until the detector baseline is stable. Start the instrument detection sequence (push to "start" button) and immediately slide the sample boat into the furnace.
- 11.6 Read the concentration of carbon from the instrument.
- 11.7 If the sample result is greater than the linear working range of the instrument as calibrated, repeat the analysis from step 11.2 using a smaller amount of sample.

11.8 Run all samples in duplicate and report the average value.

12.0 CALCULATION:

12.1 Read the μg TOC from the instrument.

12.2 If the instrument reports TOC as mg/L, calculate the μg TOC using the following equation:

$$\mu\text{g TOC} = V_{\text{STD}} \times C$$

where:

V = Volume (mL) of calibration standard injected when calibrating the range used.

C = Sample concentration (mg/L) of TOC read from instrument.

12.3 Calculate the mg/Kg TOC using the following equation:

$$\text{mg/Kg TOC} = \frac{\mu\text{g TOC}}{\text{g sample}}$$

13.0 DATA PACKAGE DELIVERABLES:

13.1 The deliverables included in the data package for analyses depend on the ASL requested. The chart below indicates which QC sample information must be included for this method for various ASL levels

DATA PACKAGE DELIVERABLES

QC SAMPLE	ANALYTICAL SUPPORT LEVELS		
	B	C	D
Sample Results	Yes	CLP Form 1 or equivalent	CLP Form 1 or equivalent

Laboratory Control Sample (LCS)	Not Required	CLP Form 7 or equivalent	CLP Form 7 or equivalent
Method Blanks	Not Required	CLP Form 3 or equivalent	CLP Form 3 or equivalent
Initial/Continuing Cal. Blanks	Not Required	CLP Form 3 or equivalent	CLP Form 3 or equivalent
Matrix Spikes	Not Required	CLP Form 5A or equivalent	CLP Form 5A or equivalent
Duplicate Samples	Not Required	CLP Form 6 or equivalent	CLP Form 6 or equivalent
Initial Calibration	Not Required	CLP Form 2 or equivalent	CLP Form 2 or equivalent
Continuing Calibration	Not Required	CLP Form 2 or equivalent	CLP Form 2 or equivalent

A/S = As specified in method or project

CLP = USEPA Contract Lab Program Statement of Work for Inorganic Analysis (SOW No. 788)

13.2 For additional information on specific data package deliverables refer to the SOP for General Laboratory Requirements.

14.0 QUALITY CONTROL REQUIREMENTS:

The following quality control samples are required for this analysis:

14.1 Calibration standards:

14.1.1 Calibration (Standard) Curve: It is preferable, if the instrumentation allows it, to calibrate with a calibration curve. The calibration curve samples are made from a standard solution. The curve should include at least 4 standards + a calibration blank (deionized water). The standards should bracket the expected sample concentrations.

- 14.1.2 Some instruments, however, are calibrated with a single mid-range standard.
- 14.1.3 Whichever method is used to calibrate an instrument, the calibration should be verified by an ICVS (14.2).
- 14.2 Initial Calibration Verification Sample (ICVS): The ICVS must be prepared from a source different from the calibration standards. One ICVS must be run with each analytical run. On the run log, the ICVS must be identified. The ICVS may also serve as the LCS. The recommended acceptable recovery range for ICVS is 90.0 - 110.0 %.
- 14.3 Continuing Calibration Verification Sample (CCVS): A mid-range standard run after every 10 samples. Use the same CCVS throughout the run. The recommended acceptable recovery range is 90.0 - 110.0 %. If a CCVS exceeds this range, stop analyses, determine the problem, recalibrate and verify the curve, and rerun all samples run since the last acceptable CCVS.
- 14.4 Initial and Continuing Calibration Blanks (ICB and CCB): Reagent blank which is used to establish that the system is free from contamination. An ICB should be run immediately after the initial calibration. CCBs are run immediately after CCVSs. ICB and CCB values should be below the reporting limit. If the blanks are positive, stop analysis and check for contamination. The system may have to be recalibrated.
- 14.5 Laboratory Control Sample (LCS): 1 per analytical batch. May be the ICVS.
- 14.6 Matrix Spike (MS): 1 per 20 samples.
- 14.7 Duplicates: 1 per 20 samples.
- 14.8 For additional information on QA Samples, refer to the SOP for General Laboratory Requirements.

15.0 METHOD VALIDATION:

Each analyst must make an initial, one-time demonstration of the ability to generate acceptable accuracy and precision with this method.

16.0

REFERENCES:

- 16.1 Dohrmann DC-80 Total Organic Carbon Systems Manual,
Edition 11, July, 1986.
- 16.2 Procedures for Handling and Chemical Analysis of
Sediment and Water Samples, EPA/Corps. of Engineers,
EPA/CE -81-1, May, 1981.
- 16.3 Methods for Chemical Analysis of Water and Wastes,
USEPA, PB84-128677, March 1983, Method 415.1

CHAPTER SEVEN

INTRODUCTION AND REGULATORY DEFINITIONS

7.1 IGNITABILITY

7.1.1 Introduction

This section discusses the hazardous characteristic of ignitability. The regulatory background of this characteristic is summarized, and the regulatory definition of ignitability is presented. The two testing methods associated with this characteristic, Methods 1010 and 1020, can be found in Chapter Eight.

The objective of the ignitability characteristic is to identify wastes that either present fire hazards under routine storage, disposal, and transportation or are capable of severely exacerbating a fire once started.

7.1.2 Regulatory Definition

The following definitions have been taken verbatim from the RCRA regulations (40 CFR 261.21).

Characteristics Of Ignitability Regulation

A solid waste exhibits the characteristic of ignitability if a representative sample of the waste has any of the following properties:

1. It is a liquid, other than an aqueous solution, containing <24% alcohol by volume, and it has a flash point <60°C (140°F), as determined by a Pensky-Martens Closed Cup Tester, using the test method specified in ASTM Standard D-93-79 or D-93-80, or a Setaflash Closed Cup Tester, using the test method specified in ASTM standard D-3278-78, or as determined by an equivalent test method approved by the Administrator under the procedures set forth in Sections 260.20 and 260.21. (ASTM standards are available from ASTM, 1916 Race Street, Philadelphia, PA 19103.)
2. It is not a liquid and is capable, under standard temperature and pressure, of causing fire through friction, absorption of moisture, or spontaneous chemical changes and, when ignited, burns so vigorously and persistently that it creates a hazard.
3. It is an ignitable compressed gas, as defined in 49 CFR 173.300 and as determined by the test methods described in that regulation or by equivalent test methods approved by the Administrator under Sections 260.20 and 260.21.
4. It is an oxidizer, as defined in 49 CFR 173.151.

[illegible]

From: Joe Basil _____ (OFFICE)
Date: 10/21/91 Project No. _____
Re: _____

Ignitability

The testing of a solid for ignitability is defined under SW 846 Chapter Seven, under Regulatory Definition (7.1.2) paragraph two. This defines solid to be ignitable in part when it ~~it~~ is ignited it burns or vigorously and persistently that it creates a hazard. EPA says that ~~this~~ is a judgement call; the analyst should use prudence in determining if the sample does burn or vigorously and persistently that it could create a hazard.

**CH2M HILL
MONTGOMERY LABORATORY
STANDARD OPERATING PROCEDURES
WET CHEMISTRY DEPARTMENT**

**METHOD FOR TOTAL CYANIDE ANALYSIS IN SOIL/SEDIMENT
CYANIDE, TOTAL (IN SEDIMENTS)**

Method 335.2 CLP-M* (Titrimetric; Manual Spectrophotometric; Semi-Automated Spectrophotometric)

1. SCOPE AND APPLICATION

- 1.1 This method applies to the determination of cyanide in sediments and other solids.
- 1.2 The detection limit depends upon the weight of sample taken for analysis.

2. SUMMARY OF METHOD

- 2.1 The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by volumetric titration or colorimetrically.
- 2.2 In the colorimetric measurement, the cyanide is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color forms upon the addition of pyridine-barbituric acid reagent. The absorbance is read at 578 nm. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.
- 2.3 The titrimetric measurement uses a standard solution of silver nitrate to titrate cyanide in the presence of a silver sensitive indicator.

3. DEFINITIONS

- 3.1 Cyanide is defined as cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

*CLP-M Modified for the Contract Laboratory Program

4. SAMPLE HANDLING AND PRESERVATION

- 4.1 Samples must be stored at 4°C(±2°C) and must be analyzed within the holding time specified in Exhibit D, Section II.
- 4.2 Samples are not dried before analysis. A separate percent solid determination must be made in accordance with the procedure in Part F.

5. INTERFERENCES

- 5.1 Interferences are eliminated or reduced by using the distillation procedure described in Procedure 8.1.
- 5.2 Sulfides adversely affect the colorimetric and titration procedures.
- 5.3 The presence of surfactants may cause the sample to foam during refluxing. If this occurs, adding an agent such as DOW Corning 544 antifoam agent will prevent the foam from collecting in the condenser. Fatty acids will distill and form soaps under the alkaline titration conditions, making the end point almost impossible to detect. When this occurs, one of the spectrophotometric methods should be used.

6. APPARATUS

- 6.1 Reflux distillation apparatus such as shown in Figure 1. The boiling flask should be 1 liter in size with an inlet tube and provision for a condenser.
- 6.2 Microburet, 5.0 ml (for titration)
- 6.3 Spectrophotometer suitable for measurements at 578 nm with a 1.0 cm cell or larger
- 6.4 Lachat QuikChem Automated Flow Injection Analyzer which includes:
 - 6.4.1 Automatic Sampler
 - 6.4.2 Proportioning Pump
 - 6.4.3 Injection Valve Module with a 150 cm 0.8 mm i.d. sample loop
 - 6.4.4 Flow Cell, 10 mm, 80 uL
 - 6.4.5 Interference Filter Wavelength, 570 nm
 - 6.4.6 Heater Module
 - 6.4.7 Reaction Module 10-204-00-1-A

7. REAGENTS

7.1 Distillation and Preparation Reagents

- 7.1.1 Sodium hydroxide solution 0.25N. Dissolve 20 g of NaOH in distilled water, and dilute to 2 liters with distilled water.
- 7.1.2 Cadmium carbonate: powdered
- 7.1.3 Ascorbic acid: crystals
- 7.1.4 Sulfuric acid: concentrated
- 7.1.5 Magnesium chloride solution: Weigh 510 g of $\text{MgCl}_2 \times 6\text{H}_2\text{O}$ into a 1,000 ml flask, dissolve, and dilute to 1 liter with distilled water.

7.2 Stock Standards and Titration Reagents

- 7.2.1 Stock cyanide solution: Dissolve 2.51 g of KCN and 2 g KOH in 1 liter of distilled water. Standardize with 0.0192 N AgNO_3 .
- 7.2.2 Standard cyanide solution, intermediate: Dilute 50.0 ml of stock (1 ml = 1 mg CN) to 1,000 ml with distilled water (1 ml = 50.0 ug).
- 7.2.3 Standard silver nitrate solution, 0.0192 N: Prepare by crushing approximately 5 g AgNO_3 crystals and drying to constant weight at 40°C. Weight out 3.2647 g of dried AgNO_3 , dissolve in distilled water, and dilute to 1,000 ml (1 ml = 1 mg CN).
- 7.2.4 Rhodanine indicator: Dissolve 20 mg of p-dimethyl-aminobenzalrhodanine in 100 ml acetone

7.3 Manual Spectrophotometric Reagents

- 7.3.1 Sodium dihydrogenphosphate, 1 M: Dissolve 138 g of $\text{NaH}_2\text{PO}_4 \times \text{H}_2\text{O}$ in 1 liter of distilled water. Refrigerate this solution.
- 7.3.2 Chloramine-T solution: Dissolve 1.0 g of white, water soluble Chloramine-T in 100 ml of distilled water and refrigerated until ready to use. Prepare fresh daily.
- 7.3.3 Color reagent
 - 7.3.3.1 Pyridine-barbituric acid reagent: Place 15 g of barbituric acid in a 250 ml volumetric flask and add just enough distilled water to wash the sides of the flask and wet the barbituric acid. Add 75 ml of pyridine and mix. Add 15

ml of HCl (sp gr 1.19), mix, and cool to room temperature. Dilute to 250 ml with distilled water and mix. This reagent is stable for approximately 6 months if it is stored in a cool, dark place.

7.4 Semi-Automated Spectrophotometric Reagents

- 7.4.1 Chloramine-T solution: Dissolve 0.40 g of chloramine-T in distilled water and dilute to 100 mL. Prepare fresh daily.
- 7.4.2 Phosphate buffer: Dissolve 138 g of $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ in distilled water and dilute to 1 liter. Add 0.5 mL of Brij-35 (available from Technicon). Store at 4°C ($\pm 2^\circ\text{C}$).
- 7.4.3 Pyridine-barbituric acid solution: Transfer 15 g of barbituric acid into a 1 liter volumetric flask. Add about 100 mL of distilled water and swirl the flask. Add 74 mL of pyridine and mix. Add 15 mL of concentrated HCl and mix. Dilute to about 900 mL with distilled water and mix until the barbituric acid is dissolved. Dilute to 1 liter with distilled water. Store at 4°C ($\pm 2^\circ\text{C}$).
- 7.4.4 Sampler wash: Dissolve 10 g of NaOH in distilled water and dilute to 1 liter.

8. PROCEDURE

8.1 Distillation

- 8.1.1 Accurately weigh a representative 1 to 5 g portion of wet sample and transfer it to a boiling flask. Add 500 ml of distilled water. Shake or stir the sample so that it is dispersed.
- 8.1.2 Add exactly 100 ml of sodium hydroxide (7.1.1) to the absorbing tube. Connect the boiling flask, condenser, absorber, and trap in the train.
- 8.1.3 Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately one bubble of air per second enters the boiling flask through the air inlet tube.

NOTE: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It will be necessary to readjust the air rate occasionally to prevent the solution in the boiling flask from backing up into the air inlet tube.

- 8.1.4 Slowly add 25 ml of concentrated sulfuric acid (7.1.4) through the air inlet tube. Rinse the tube with distilled water and allow the airflow to

mix the flask contents for 3 minutes. Pour 20 ml of magnesium chloride solution (7.1.5) into the air inlet and wash down with a stream of water.

8.1.5 Heat the solution to boiling, taking care to prevent the solution from backing up and overflowing into the air inlet tube. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect absorber and close off the vacuum source.

8.2 Titrimetric Determination (Option A)

8.2.1 If the sample contains more than 1 mg of CN, transfer the distillate, or a suitable aliquot diluted to 250 ml, to a 500 ml Erlenmeyer flask. Add 10-12 drops of the benzalrhodanine indicator.

8.2.2 Titrate with standard silver nitrate to the first change in color from yellow to brownish-pink. Titrate a distilled water blank using the same amount of sodium hydroxide and indicator as in the sample.

8.2.3 The analyst should familiarize himself with the end point of the titration and the amount of indicator to be used before actually titrating the samples. A 5 or 10 ml microburet may be used conveniently to obtain a more precise titration.

8.3 Manual Spectrophotometric Determination (Option B)

8.3.1 Withdraw 50 ml or less of the solution from the absorber tube and transfer to a 100 ml volumetric flask. If less than 50 ml is taken, dilute to 50 ml with 0.25 N sodium hydroxide solution (7.2.6). Add 15.0 ml of sodium phosphate solution (7.3.2) and mix.

8.3.1.1 Pyridine-barbituric acid method: Add 2 ml of Chloramine-T (7.3.2) and mix. After 1 to 2 minutes, add 5 ml of pyridine-barbituric acid solution (7.3.3.1) and mix. Dilute to mark with distilled water and mix again. Allow 8 minutes for color development, then read absorbance at 578 nm in a 1 cm cell within 15 minutes.

8.3.2 Prepare a minimum of 5 standards and a blank by pipetting suitable volumes of standard solution into 100 ml volumetric flasks.

NOTE: One calibration standard must be made at the CRDL. To each standard, add 50 ml of 0.25 N sodium hydroxide. Standards must bracket the concentrations of the sample. If dilution is required, use the blank solution.

As an example, standard solutions could be prepared as follows:

<u>ul of Standard Solution</u>	<u>conc. ug CN</u>
<u>7.2.2</u>	
0	Blank
50	5
100	10
250	25
500	50
1,000	100

8.3.2.1 It is not imperative that all standards be distilled in the same manner as the samples. At least one standard (mid-range) must be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable. If the distilled standard does not agree within $\pm 15\%$ of the undistilled standards, the operator should find and correct the cause of the apparent error before proceeding.

8.3.2.2 Prepare a standard curve by plotting absorbance of standard vs. cyanide concentrations.

8.4 Semi-Automatic Spectrophotometric Determination (Option C)

8.4.1 Set up the manifold as shown in manifold diagram. Pump the reagents through the system until a steady baseline is obtained.

8.4.2 Calibration standards: Prepare a blank and at least five calibration standards over the range of the analysis. One calibration standard must be at the CRDL. For a working range of 0-200 ug/L, the following standards may be used:

8.4.2.1 It is not imperative that all standards be distilled in the same manner as the samples. At least one standard (mid-range) must be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable. If the distilled standard does not agree within ± 15 percent of the undistilled standards, the operator should find and correct the cause of the apparent error before proceeding.

<u>uL Standard Solution (7.2.2) diluted to 100 ml</u>	<u>Concentration ug CN/L</u>
0	0
50	2.5
100	5
200	10
500	25
1,000	50
2,000	100

Add 1.0 g of NaOH to each standard. Store at 4°C ($\pm 2^\circ\text{C}$).

8.4.3 Place calibration standards, blanks, and control standards in the sampler tray, followed by distilled samples, distilled duplicates, distilled standards, distilled spikes, and distilled blanks.

8.4.4 Set Injection Timing With:

- 8.4.4.1 Pump speed: 35
- 8.4.4.2 Cycle period: 40 s
- 8.4.4.3 Sample Loop Length: 150 cm
- 8.4.4.4 Load period: 20 s
- 8.4.4.5 Inject period: 20 s
- 8.4.4.6 Inject to start of peak period: 25 s
- 8.4.4.7 Inject to end of peak period: 61 s

8.4.5 Set System IV Gain: 340 x 1

8.4.6 System Operation

- 8.4.6.1 Inspect modules for proper connections.
- 8.4.6.2 Turn on power to all modules. Allow heater to warm up to 60°C.
- 8.4.6.3 Place reagent transmission lines into proper containers. Raise tension levers on pump tube cassettes.
- 8.4.6.4 Pump system until a stable baseline is attained.
- 8.4.6.5 Set baseline. If necessary, manually inject a high standard to set gain on colorimeter.
- 8.4.6.6 Program data system to initial parameters or those empirically determined.

- 8.4.6.7 Place calibration standards and blank in sample tray in descending order of concentration followed by unknowns, and check standards.
- 8.4.6.8 At end of run, place all transmission lines in water, flush system and pump dry.
- 8.4.6.9 Turn off pump, all modules, and release pump tube cassettes.

9. CALCULATIONS

- 9.1 A separate determination of percent solids must be performed (see Part F).
- 9.2 The concentration of cyanide in the sample is determined as follows:
- 9.2.1 (Titration)

$$\text{CN, mg/kg} = \frac{(A - B) \times \frac{100 \text{ ml}}{\text{ml aliquot titrated}} \times 1,000 \text{ g/kg}}{C \times \frac{\% \text{ solids}}{100}}$$

Where:

A	=	ml of AgNO ₃ for titration of sample (1 ml = 1 mg Ag)
B	=	ml of AgNO ₃ for titration of blank (1 ml = 1 mg Ag)
C	=	wet weight of original sample in g (See 8.1.1)

And: 100 ml = volume of distillate (See 8.1.6)
 1,000 g/kg = conversion factor g to kg
 ml aliquot titrated (See 8.2.1)
 % solids (See Part F)

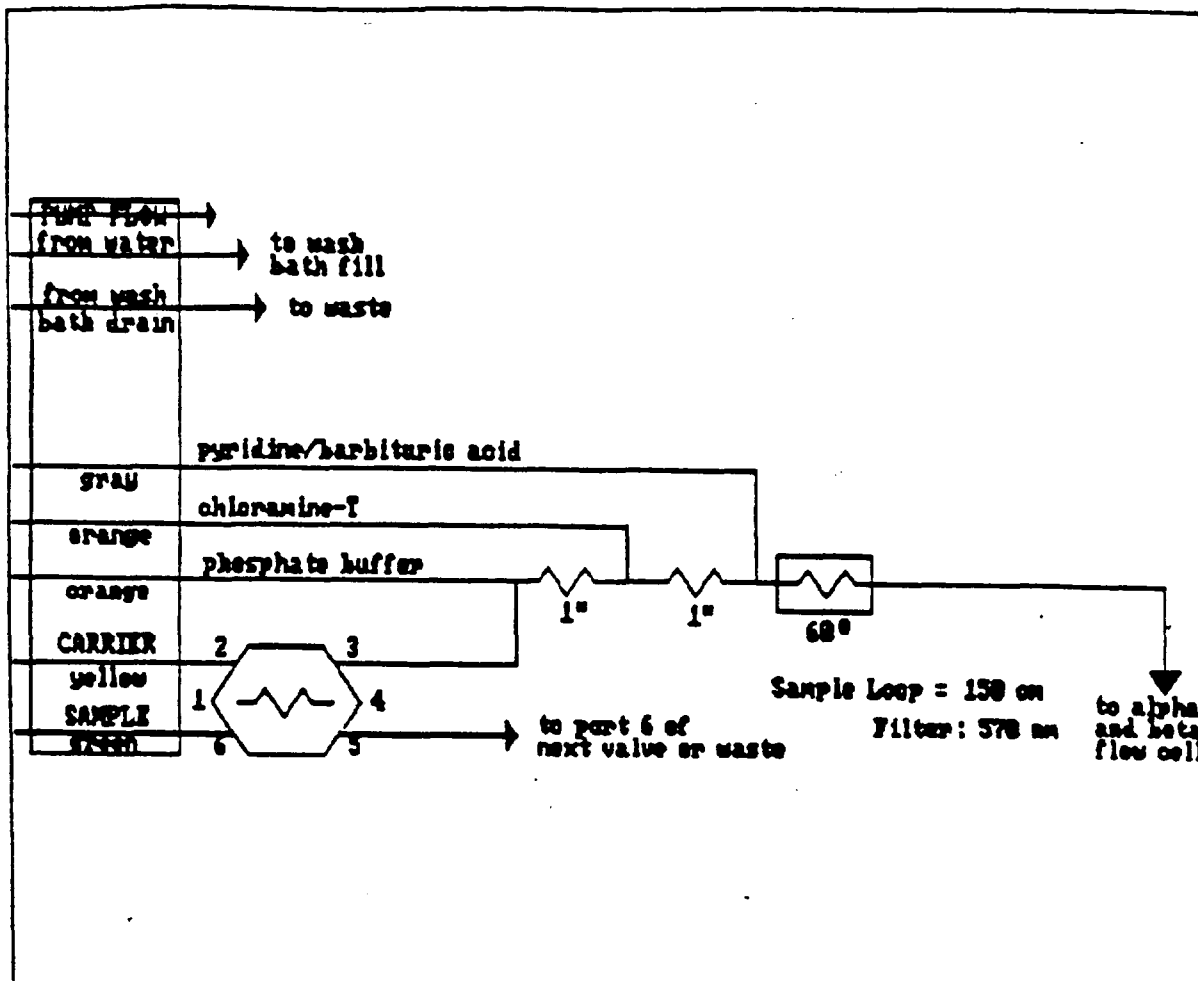
10.0 Quality Control Samples: For Cyanide analyses, the following control samples are included on the bench sheet and should be run with each batch of samples:

- method blank
- QC check sample
- duplicate samples
- matrix spike/duplicate

Acceptance limits for these quality control samples are as follows:

- method blank - if the analyte of interest is detected in the method blank, the samples and blank must be redistilled and reanalyzed. One method blank must be distilled with each distillation batch.
- QC check sample - The spreadsheet has an area for entering data from the QC check sample. True value is given and the % recovery is calculated. This is charted on a control chart and statistical information is generated. The recovery on the QC sample must be within $\pm 3S$ for acceptance or within $\pm 15\%$ of the True value, whichever is the most stringent. This LCS must be distilled with each distillation batch and must be on EPA traceable (like ICV-6). This LCS is done in addition to the distilled standard. When the QC recovery is outside this range, the system must be checked, a new QC sample made up, and the associated batch of samples must be re-distilled and re-analyzed. This must be documented on a corrective action report.
- duplicate samples - Generally an RPD of 20 is considered the outside limit. The spreadsheet has an area for entry of duplicate analysis data. This will be charted after each analytical run. Acceptance limits are RPD inside $+ 3S$.
- matrix spike - a sample spiked with 1.00 mL of the intermediate cyanide standard (7.2.2), the matrix spike recovery must be within $\pm 15\%$ of the true spike amount.

Manifold Diagram



CARRIER is 0.25 M sodium hydroxide, Reagent 1.

1"	is	70.0	cm of tubing on a 1 in coil support
2"	is	135	cm of tubing on a 2 in coil support
2.5"	is	168	cm of tubing on a 2.5 in coil support
3"	is	202	cm of tubing on a 3 in coil support
4"	is	255	cm of tubing on a 4 in coil support
8"	is	550	cm of tubing on a 8 in coil support

Heated tubing is shown inside a box with the temperature next to the box. heated tubing is 650 cm unless otherwise specified.

All manifold tubing is 0.8 mm (0.032 in) i.d. This is 5.2 uL/cm.

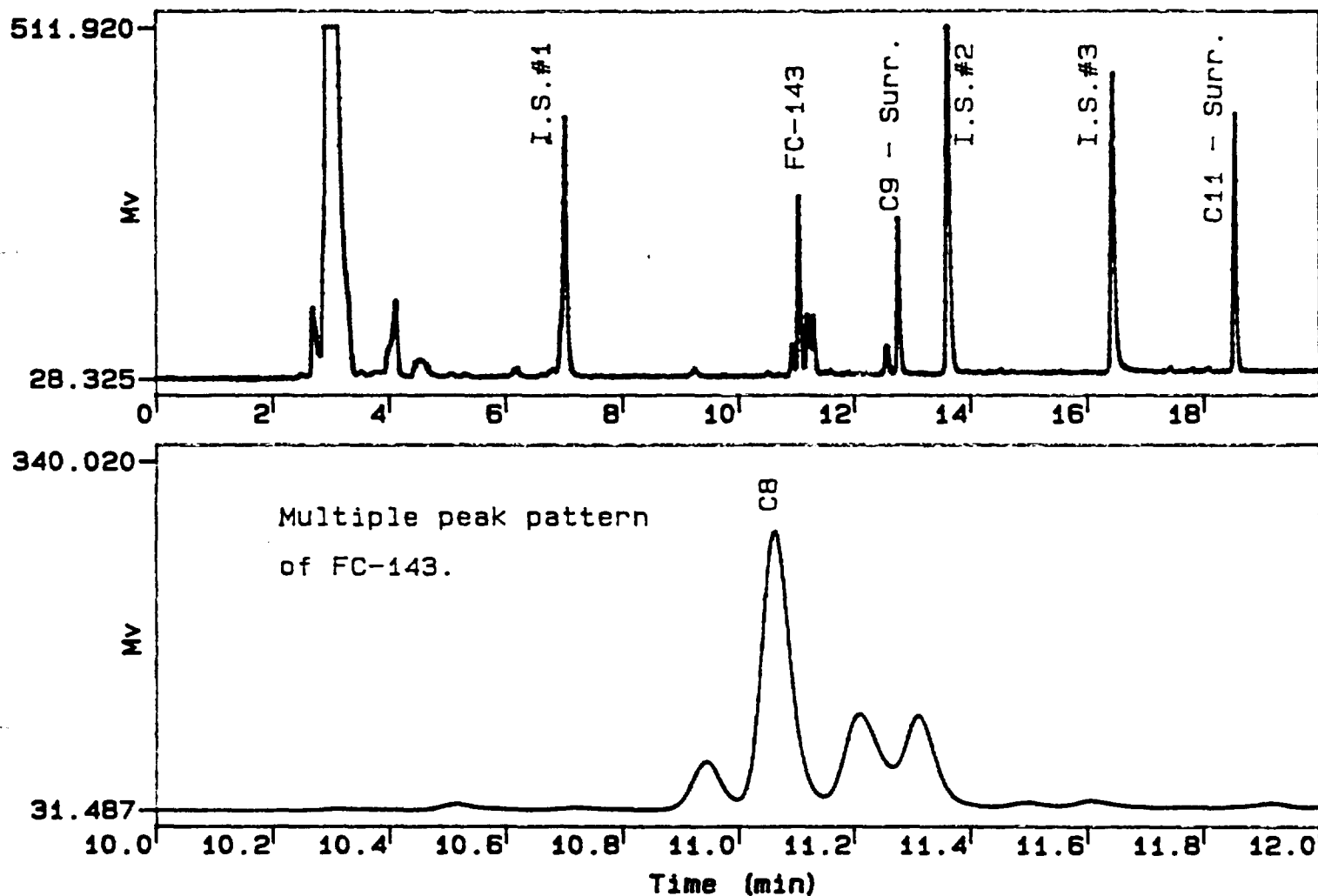


Figure 3. GC/ECD chromatogram of instrument standard at 1 ng/mL and multiple peak pattern of FC-143. Fused silica capillary column: Rtx-200 (Restek Corporation), 30m x 0.32mm I.D., 0.5 μ m film thickness. Column program: 50°C for 2 minutes to 90°C at 5°C/minute then to 280°C at 10°C/minute and hold .2 minutes. Injector zone: 200°C. Detector zone: 320°C. Helium carrier inlet pressure = 5.5psig. 1 μ L splitless injection, split on at 0.5 minutes.

**CH2M HILL
MONTGOMERY LABORATORY
STANDARD OPERATING PROCEDURES
WET CHEMISTRY DEPARTMENT**

METHOD FOR TOTAL SULFIDE ANALYSIS IN SOIL/SEDIMENT

SULFIDE, TOTAL (IN SEDIMENTS)

Method 376.1 Modified (Titrimetric, Iodine)

1. SCOPE AND APPLICATION

- 1.1 This method applies to the measurement of total sulfides in sediments and sludges.
- 1.2 Acid-insoluble sulfides are not measured by this test. (Copper sulfide is the only common sulfide in this class.)
- 1.3 This method is suitable for the measurement of sulfide in concentrations above 4 mg/kg.

2. SUMMARY OF METHOD

- 2.1 Excess iodine is added to the nonfilterable portion of a sample that has been treated with zinc acetate to produce zinc sulfide. This iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is backtitrated with sodium thiosulfate or phenylarsine oxide.

3. COMMENTS

- 3.1 Reduced sulfur compounds such as sulfite, thiosulfate and hydrosulfite that decompose in acid may yield erratic results. Also, volatile iodine-consuming substances will give high results. These interferences are eliminated in Sections 6.3 and 6.4.

4. APPARATUS

Ordinary Laboratory Glassware

5. REAGENTS

- 5.1 Hydrochloric acid, HCl, 6 N

- 5.2 Standard iodine solution, 0.0250 N: Dissolve 20 to 25 g KI in a little water in a liter volumetric and add 3.2 g iodine. Allow to dissolve. Dilute to 1 liter and standardize against 0.0250 N sodium thiosulfate or phenylarsine oxide using a starch indicator.
- 5.3 Sodium thiosulfate 0.0250 N: commercially available.
- 5.4 Starch indicator: commercially available.
- 5.5 Sodium hydroxide, 0.25 N: Dissolve 10 g NaOH in a 1 liter volumetric flask, bring to volume, and mix.

6.0 PROCEDURE

- 6.1 Place 10.0 g of sample in beaker. Slowly add 50 ml distilled water without introducing air, then add 1 ml of 0.25 N NaOH followed by 0.4 g of zinc acetate.
- 6.2 Add a spin bar and slowly mix without vortexing for 30 minutes.
- 6.3 Filter entire sample through glass fiber filter paper (Whatman 934 AH).
- 6.4 Rinse sample with 3 portions of 50 ml of H₂O.
- 6.5 Transfer unfilterable sample and filter to a 500 ml erlenmeyer flask; then add 200 ml distilled H₂O.
- 6.6 Add excess iodine solution (5.2) and starch indicator (5.4).
- 6.7 Add 2 ml of 6 N HCl (5.1).
- 6.8 If iodine color disappears, add more iodine until the color remains for more than 5 minutes after mixing.
- 6.9 Titrate with .025 N sodium thiosulfate until blue color disappears.

7. CALCULATIONS

- 7.1 Sample concentration

$$\text{mg/kg sulfide} = \frac{400 (A - B) \times 100}{\text{g sample} \times \% \text{ solids}}$$

Where:

- A = ml of 0.025 N standard iodine solution (5.2)
 B = ml of 0.025 N standard reducing sodium thiosulfate

7.2 Determination of amount spiked

$$g \text{ of } Na_2S \cdot 9H_2O \times \frac{32.06}{240.18} \times \frac{1000}{g \text{ sample}} \times \frac{100\%}{\% \text{ solids sample}} \times \frac{1,000mg}{1g} = mg/kg S^-$$

8. QUALITY CONTROL

The following control samples are to be included with each analytical batch of samples:

- Method blank
- Blank spike
- Matrix spike
- Duplicate sample

Method blank

10.0 g of muffled sand (muffled at 550°C for 4 hours) to be used as the method blank. If the analyte of interest is detected in the method blank, the analytical batch must be reanalyzed and the problem investigated.

Blank spike

10.0 g of muffled sand spiked with $Na_2S \cdot 9H_2O$ dry crystals (between 0.010 g to 0.006 g). The percent recovery must be within ± 3 standard deviations and within 10 percent of the true value.

Matrix spike

10.0 g of sample spiked with $Na_2S \cdot 9H_2O$ dry crystals. The percent recovery must be within 10 percent of the true value.

Duplicate sample

The results of duplicate samples must be within 20 RPD.

7.3 REACTIVITY

7.3.1 Introduction

The regulation in 40 CFR 261.23 defines reactive wastes to include wastes that have any of the following properties: (1) readily undergo violent chemical change; (2) react violently or form potentially explosive mixtures with water; (3) generate toxic fumes when mixed with water or, in the case of cyanide- or sulfide-bearing wastes, when exposed to mild acidic or basic conditions; (4) explode when subjected to a strong initiating force; (5) explode at normal temperatures and pressures; or (6) fit within the Department of Transportation's forbidden explosives, Class A explosives, or Class B explosives classifications.

This definition is intended to identify wastes that, because of their extreme instability and tendency to react violently or explode, pose a problem at all stages of the waste management process. The definition is to a large extent a paraphrase of the narrative definition employed by the National Fire Protection Association. The Agency chose to rely on a descriptive, prose definition of reactivity because the available tests for measuring the variegated class of effects embraced by the reactivity definition suffer from a number of deficiencies.

7.3.2 Regulatory Definition

7.3.2.1 Characteristic Of Reactivity Regulation

A solid waste exhibits the characteristic of reactivity if a representative sample of the waste has any of the following properties:

1. It is normally unstable and readily undergoes violent change without detonating.
2. It reacts violently with water.
3. It forms potentially explosive mixtures with water.
4. When mixed with water, it generates toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or to the environment.
5. It is a cyanide- or sulfide-bearing waste that, when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or to the environment. (Interim Guidance for Reactive Cyanide and Reactive Sulfide, Sections 7.3.3 and 7.3.4 below, can be used to detect the presence of cyanide and sulfide in wastes.)

6. It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.
7. It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure.
8. It is a forbidden explosive, as defined in 49 CFR 173.51, or a Class A explosive, as defined in 49 CFR 173.53, or a Class B explosive, as defined in 49 CFR 173.88.
9. A solid waste that exhibits the characteristic of reactivity, but is not listed as a hazardous waste in Subpart D, has the EPA Hazardous Waste Number of D003.

7.3.3 Interim Guidance For Reactive Cyanide

7.3.3.1 The current EPA action level is:

Total releasable cyanide: 250 mg HCN/kg waste.

7.3.3.2 Test Method to Determine Hydrogen Cyanide Released from Wastes

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to all wastes, with the condition that wastes that are combined with acids do not form explosive mixtures.

1.2 This method provides a way to determine the specific rate of release of hydrocyanic acid upon contact with an aqueous acid.

1.3 This test measures only the hydrocyanic acid evolved at the test conditions. It is not intended to measure forms of cyanide other than those that are evolvable under the test conditions.

2.0 SUMMARY OF METHOD

2.1 An aliquot of the waste is acidified to pH 2 in a closed system. The gas generated is swept into a scrubber. The analyte is quantified. The procedure for quantifying the cyanide is Method 9010, Chapter Five, starting with Step 7.3.5 of that method.

3.0 SAMPLE HANDLING AND PRESERVATION

3.1 Samples containing, or suspected of containing, sulfide or a combination of sulfide and cyanide wastes should be collected with a minimum of aeration. The sample bottle should be filled completely, excluding all head space, and stoppered. Analysis should commence as soon as possible, and samples should be kept in a cool, dark place until analysis begins.

3.2 It is suggested that samples of cyanide wastes be tested as quickly as possible. Although they can be preserved by adjusting the sample pH to 12 with strong base, this will cause dilution of the sample, increase the ionic strength, and, possibly, change other physical or chemical characteristics of the waste which may affect the rate of release of the hydrocyanic acid. Storage of samples should be under refrigeration and in the dark.

3.3 Testing should be performed in a ventilated hood.

4.0 APPARATUS AND MATERIALS (See Figure 1)

4.1 Round-bottom flask: 500-mL, three-neck, with 24/40 ground-glass joints.

4.2 Stirring apparatus: To achieve approximately 30 rpm. This may be either a rotating magnet and stirring bar combination or an overhead motor-driven propeller stirrer.

4.3 Separatory funnel: With pressure-equalizing tube and 24/40 ground-glass joint and Teflon sleeve.

4.4 Flexible tubing: For connection from nitrogen supply to apparatus.

4.5 Water-pumped or oil-pumped nitrogen gas: With two-stage regulator.

4.6 Rotometer: For monitoring nitrogen gas flow rate.

5.0 REAGENTS

5.1 Sulfuric acid, 0.005 M: Add 2.8 mL concentrated H_2SO_4 to Type II water and dilute to 1 L. Withdraw 100 mL of this solution and dilute to 1 L to make the 0.005 M H_2SO_4 .

5.2 Cyanide reference solution: Dissolve approximately 2.5 g of KOH and 2.51 g of KCN in 1 liter of distilled water. Cyanide concentration in this solution is 1 mg/mL.

5.3 NaOH solution, 1.25 N: Dissolve 50 g of NaOH in distilled water and dilute to 1 liter with distilled water.

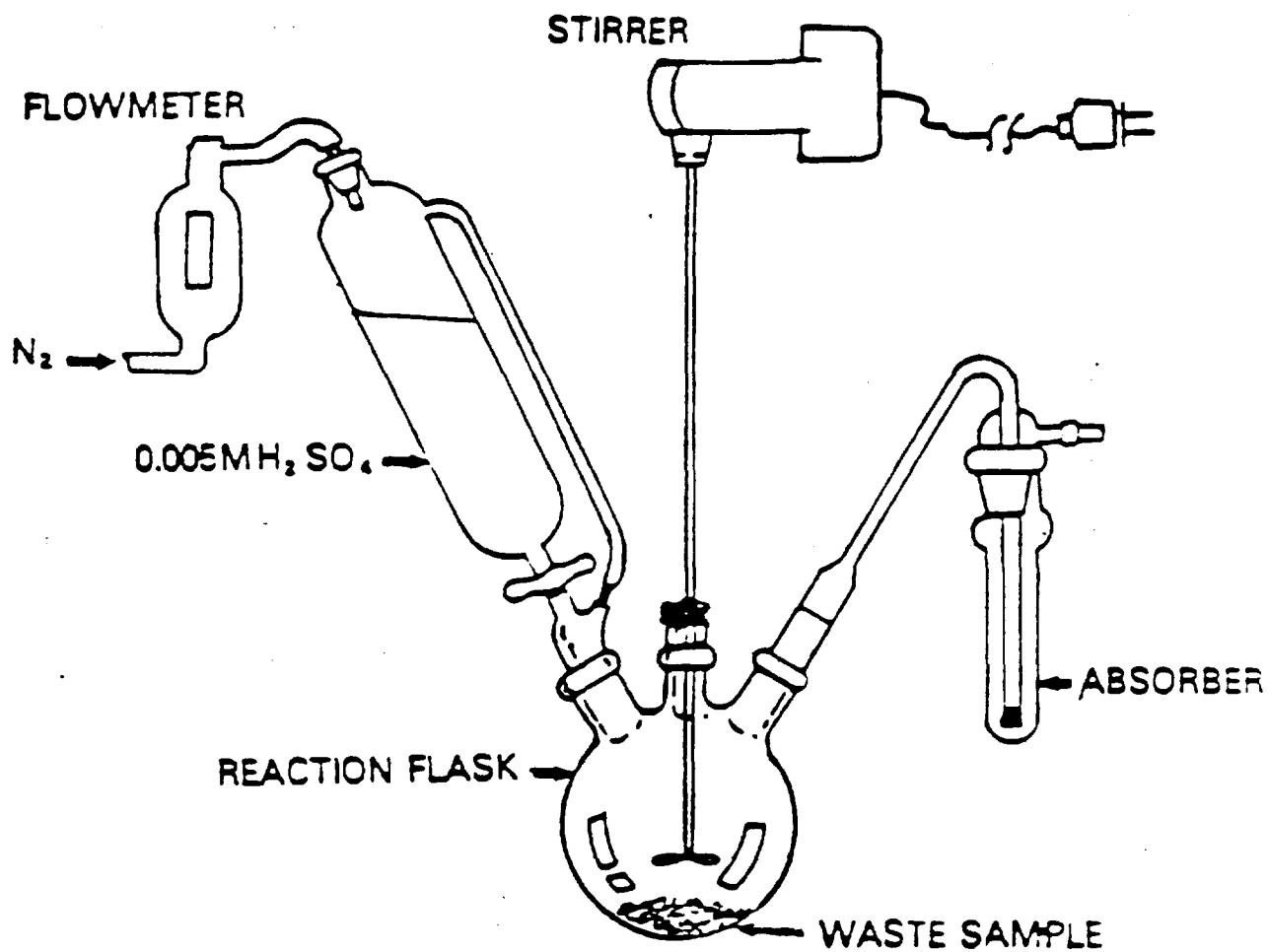


Figure 1. Apparatus to Determine Hydrogen Cyanide Released from Wastes

5.4 NaOH solution, 0.25 N: Dilute 200 mL of sodium hydroxide solution (5.3) to 1 liter with distilled water.

5.5 Stock cyanide solution, 1 mg/mL: Dissolve 2.51 g of KCN and 2 g of KOH in 1 liter of distilled water. Standardize with 0.0192 N AgNO₃. Dilute to appropriate concentration so that 1 mL = 1 mg CN.

5.6 Intermediate cyanide solution: Dilute 50 mL of stock solution to 1 liter with distilled water.

5.7 Standard cyanide solution, 5 mg/L: Prepare fresh daily by diluting 100 mL of intermediate solution to 1 liter with distilled water, and store in a glass-stoppered bottle.

5.8 Silver nitrate solution: Prepare by crushing approximately 5 g of AgNO₃ crystals and drying to constant weight at 40°C. Weigh 3.3 g of dried AgNO₃, dissolve in distilled water, and dilute to 1 liter.

5.9 Rhodanine indicator: Dissolve 20 mg of p-dimethylaminobenzal-rhodanine in 100 mL of acetone.

5.10 Methyl red indicator: Prepare by dissolving 0.02 g methyl red in 60 mL of distilled water and 40 mL of acetic acid.

6.0 SYSTEM CHECK

6.1 The operation of the system can be checked and verified using the cyanide reference solution (Paragraph 5.2). Perform the procedure using the reference solution as a sample and determine the percent recovery. A recovery of 50% is adequate to demonstrate proper system operation.

7.0 PROCEDURE

7.1 Add 500 mL of 0.25 N NaOH solution to a calibrated scrubber and dilute with distilled water to obtain an adequate depth of liquid.

7.2 Close the system and adjust the flow rate of nitrogen, using the rotometer. Flow should be 60 mL/min.

7.3 Add to the system 10 g of the waste to be tested.

7.4 With the nitrogen flowing, add enough acid to fill the system half full. While starting the 30-min test period.

7.5 Begin stirring while the acid is entering the round-bottom flask.

7.6 After 30 min, close off the nitrogen and disconnect the scrubber. Determine the amount of cyanide in the scrubber by Method 9010, Chapter Five, starting with Paragraph 7.3.5. of the method.

8.0 CALCULATIONS

8.1 Determine the specific rate of release of HCN, using the following parameters:

A = Concentration of HCN in scrubber (mg/L)
(This is obtained from Method 9010.)

L = Volume of solution in scrubber (L)

W = Weight of waste used (kg)

S = Time of measurement = Time N₂ stopped - Time
N₂ started (sec)

$$R = \text{specific rate of release} = \frac{A \cdot L}{W \cdot S}$$

$$\text{Total available HCN (mg/kg)} = R \times 1,800.$$

7.3.4 Interim Guidance For Reactive Sulfide

7.3.4.1 The current EPA action level is:

Total releasable sulfide: 500 mg H₂S/kg waste.

7.3.4.2 Test Method to Determine Hydrogen Sulfide Released from Wastes

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to all wastes, with the condition that waste that are combined with acids do not form explosive mixtures.

1.2 This method provides a way to determine the specific rate of release of hydrogen sulfide upon contact with an aqueous acid.

1.3 This procedure releases only the evolved hydrogen sulfide at the test conditions. It is not intended to measure forms of sulfide other than those that are evolvable under the test conditions.

2.0 SUMMARY OF METHOD

2.1 An aliquot of the waste is acidified to pH 2 in a closed system. The gas generated is swept into a scrubber. The analyte is quantified. The procedure for quantifying the sulfide is given in Method 9030, Chapter Five.

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Date September 1986

3.0 SAMPLE HANDLING AND PRESERVATION

3.1 Samples containing, or suspected of containing, sulfide wastes should be collected with a minimum of aeration. The sample bottle should be filled completely, excluding all head space, and stoppered. Analysis should commence as soon as possible, and samples should be kept in a cool, dark place until analysis begins.

3.2 It is suggested that samples of sulfide wastes be tested as quickly as possible. Although they can be preserved by adjusting the sample pH to 12 with strong base and adding zinc acetate to the sample, these will cause dilution of the sample, increase the ionic strength, and, possibly, change other physical or chemical characteristics of the waste which may affect the rate of release of the hydrogen sulfide. Storage of samples should be under refrigeration and in the dark.

3.3 Testing should be performed in a ventilated hood.

4.0 APPARATUS (See Figure 2)

4.1 Round-bottom flask: 500-mL, three-neck, with 24/40 ground-glass joints.

4.2 Stirring apparatus: To achieve approximate 30 rpm. This may be either a rotating magnet and stirring bar combination or an overhead motor-driven propeller stirrer.

4.3 Separatory funnel: With pressure-equalizing tube and 24/40 ground-glass joint and Teflon sleeve.

4.4 Flexible tubing: For connection from nitrogen supply to apparatus.

4.5 Water-pumped or oil-pumped nitrogen gas: With two-stage regulator.

4.6 Rotometer: For monitoring nitrogen gas flow rate.

4.7 Detector tube for sulfide: Industrial-hygiene type (100-2,000 ppm range).

5.0 REAGENTS

5.1 Sulfuric acid, 0.005 M: Add 2.8 mL concentrated H_2SO_4 to Type II water and dilute to 1 L. Withdraw 100 mL of this solution and dilute to 1 L to make the 0.005 M H_2SO_4 .

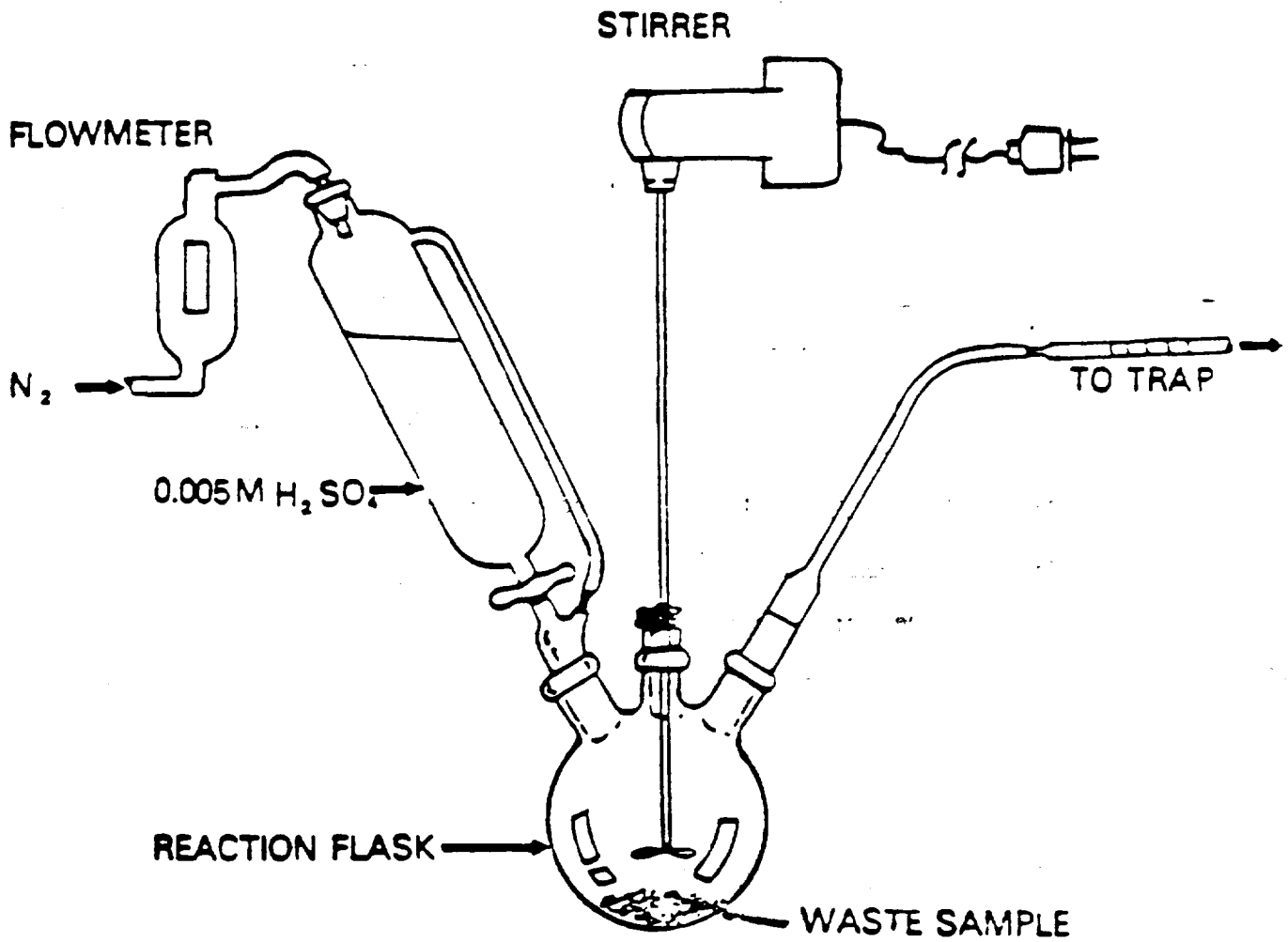


Figure 2. Apparatus to Determine Hydrogen Sulfide Released from Wastes

5.2 Sulfide reference solution: Dissolve 4.02 g of $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ in 1.0 liter of distilled water. This solution contains 680 ppm hydrogen sulfide. Dilute this stock solution to cover the analytical range required (100-680 ppm).

5.3 NaOH solution, 1.25 N: Dissolve 50 g of NaOH in distilled water and dilute to 1 liter with distilled water.

5.4 NaOH solution, 0.25 N: Dilute 200 mL of sodium hydroxide solution to 1 liter with distilled water.

6.0 SYSTEM CHECK

6.1 The operation of the system can be checked and verified using the sulfide reference solution (Paragraph 5.2). Perform the procedure using the reference solution as a sample and determine the percent recovery. A recovery of 50% is adequate to demonstrate proper system operation.

7.0 PROCEDURE

The procedure employs a scrubber solution with wet method quantification.

7.1 Add 500 mL of 0.25 N NaOH solution to a calibrated scrubber and dilute with distilled water to obtain an adequate depth of liquid.

7.2 Assemble the system and adjust the flow rate of nitrogen, using the rotometer. Flow should be 60 mL/min.

7.3 Add to the system 10 g of the waste to be tested.

7.4 With the nitrogen flowing, add enough acid to fill the system half full, while starting the 30-min test period.

7.5 Begin stirring while the acid is entering the round-bottom flask.

7.6 After 30 min, close off the nitrogen and disconnect the scrubber. Determine the amount of sulfide in the scrubber by Method 9030, Chapter 5.

7.7 Substitute the following for 7.2.3 in Method 9030: The trapping solution must be brought to a pH of 2 before proceeding. Titrate an aliquot of the trapping solution to a pH 2 end point and calculate the amount of acid needed for the 200-mL sample for analysis.

8.0 CALCULATIONS

8.1 Determine the specific rate of release of H_2S , using the following parameters:

A = Concentration of H₂S in scrubber (mg/L)
(This is obtained from Method 9030.)

L = Volume of solution in scrubber (L)

W = Weight of waste used (kg)

S = Time of experiment = Time N₂ stopped - Time
N₂ started (sec)

R = specific rate of release = $\frac{A \cdot L}{W \cdot S}$

Total available H₂S (mg/kg) = R x 1,800.

METHOD 9045

SOIL pH

1.0 SCOPE AND APPLICATION

1.1 Method 9045 is an electrometric procedure which has been approved for measuring pH in calcareous and noncalcareous soils.

2.0 SUMMARY OF METHOD

2.1 The soil sample is mixed either with Type II water or with a calcium chloride solution (see Section 5.0), depending on whether the soil is considered calcareous or noncalcareous. The pH of the solution is then measured with a pH meter.

3.0 INTERFERENCES

3.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10 , the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true pH of <1 , may give incorrectly high pH measurements.

3.2 Temperature fluctuations will cause measurement errors.

3.3 Errors will occur when the electrodes become coated. If an electrode becomes coated with an oily material that will not rinse free, the electrode can either (1) be cleaned with an ultrasonic bath, or (2) be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with water.

4.0 APPARATUS AND MATERIALS

4.1 pH Meter with means for temperature compensation.

4.2 Electrodes:

4.2.1 Calomel electrode.

4.2.2 Glass electrode.

4.2.3 A combination electrode can be employed instead of calomel or glass.

4.5 Beaker: 50-mL.

4.6 Volumetric flask: 2-Liter.

4.7 Volumetric flask: 1-Liter.

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

5.2 Primary standard buffer salts are available from the National Bureau of Standards (NBS) and should be used in situations where extreme accuracy is necessary. Preparation of reference solutions from these salts requires some special precautions and handling, such as low-conductivity dilution water, drying ovens, and carbon-dioxide-free purge gas. These solutions should be replaced at least once each month.

5.3 Secondary standard buffers may be prepared from NBS salts or purchased as solutions from commercial vendors. These commercially available solutions, which have been validated by comparison with NBS standards, are recommended for routine use.

5.4 Stock calcium chloride solution (CaCl_2), 3.6 M: Dissolve 1059 g of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ in Type II water in a 2-liter volumetric flask. Cool the solution, dilute it to volume with Type II water, and mix it well. Dilute 20 mL of this solution to 1 liter with Type II water in a volumetric flask and standardize it by titrating a 25-mL aliquot of the diluted solution with standard 0.1 N AgNO_3 , using 1 mL of 5% K_2CrO_4 as the indicator.

5.5 Calcium chloride (CaCl_2), 0.01 M: Dilute 50 mL of stock 3.6 M CaCl_2 to 18 liters with Type II water. If the pH of this solution is not between 5 and 6.5, adjust the pH by adding a little $\text{Ca}(\text{OH})_2$ or HCl . As a check on the preparation of this solution, measure its electrical conductivity. The specific conductivity should be 2.32 ± 0.08 mmho per cm at 25°C .

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.2 Samples should be analyzed as soon as possible.

7.0 PROCEDURE

7.1 Calibration:

7.1.1 Because of the wide variety of pH meters and accessories, detailed operating procedures cannot be incorporated into this method. Each analyst must be acquainted with the operation of each system and familiar with all instrument functions. Special attention to care of the electrodes is recommended.

7.1.2 Each instrument/electrode system must be calibrated at a minimum of two points that bracket the expected pH of the samples and are approximately three pH units or more apart. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer solution value.

7.2 Sample preparation and pH measurement of noncalcareous soils:

7.2.1 To 20 g of soil in a 50-mL beaker, add 20 mL of Type II water and stir the suspension several times during the next 30 min.

7.2.2 Let the soil suspension stand for about 1 hr to allow most of the suspended clay to settle out from the suspension.

7.2.3 Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant solution to establish a good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrodes into the sample solution in this manner. For combination electrodes, immerse just below the suspension.

7.2.4 If the sample temperature differs by more than 2°C from the buffer solution, the measured pH values must be corrected.

7.2.5 Report the results as "soil pH measured in water."

7.3 Sample preparation and pH measurement of calcareous soils:

7.3.1 To 10 g of soil in a 50-mL beaker, add 20 mL of 0.01 M CaCl_2 (Step 5.5) solution and stir the suspension several times during the next 30 min.

7.3.2 Let the soil suspension stand for about 30 min to allow most of the suspended clay to settle out from the suspension.

7.3.3 Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed well into the partly settled suspension and the calomel electrode will be immersed just deep enough into the clear supernatant solution to establish a good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrode into the sample solution in this manner.

7.3.4 If the sample temperature differs by more than 2°C from the buffer solution, the measured pH values must be corrected.

7.3.5 Report the results as "soil pH measured in 0.01 M CaCl_2 ".

8.0 QUALITY CONTROL

8.1 Duplicate samples and check standards should be analyzed routinely.

8.2 Electrodes must be thoroughly rinsed between samples.

9.0 METHOD PERFORMANCE

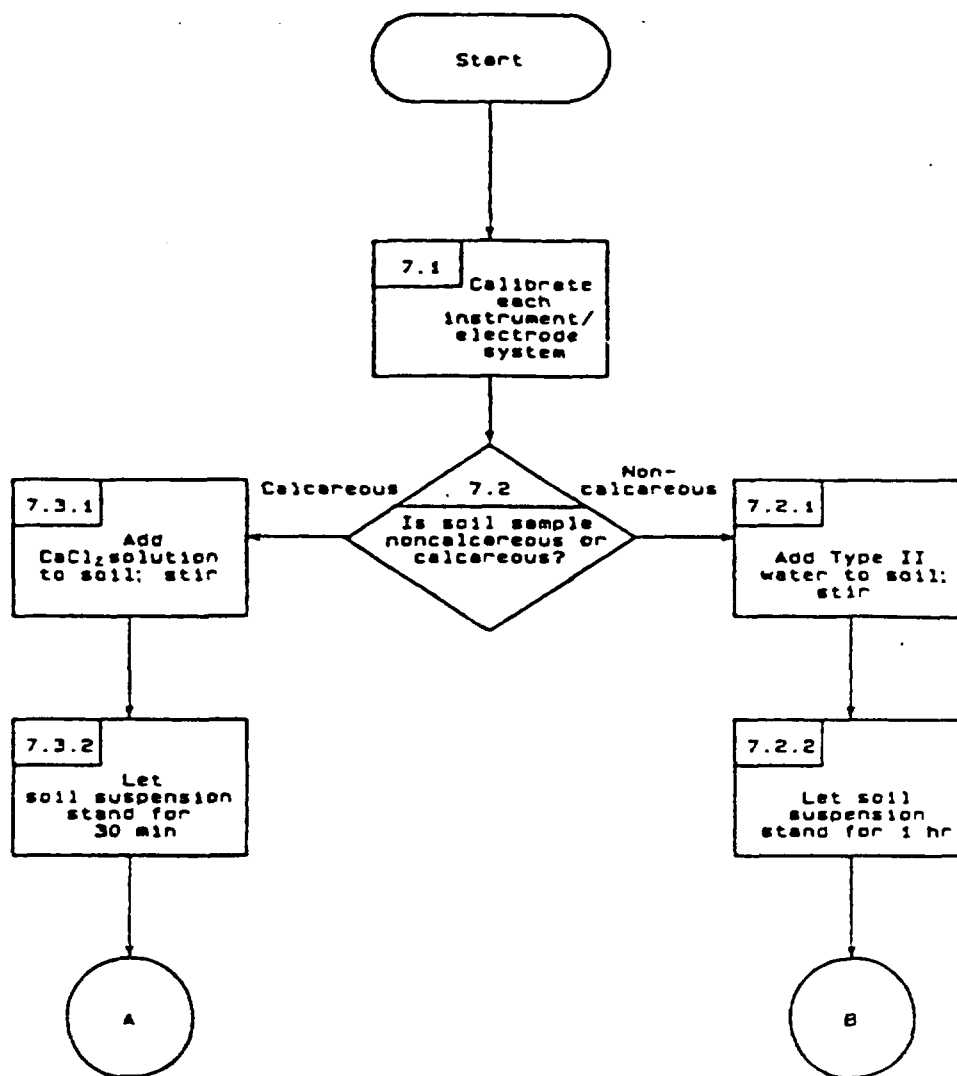
9.1 No data provided.

10.0 REFERENCES

10.1 None required.

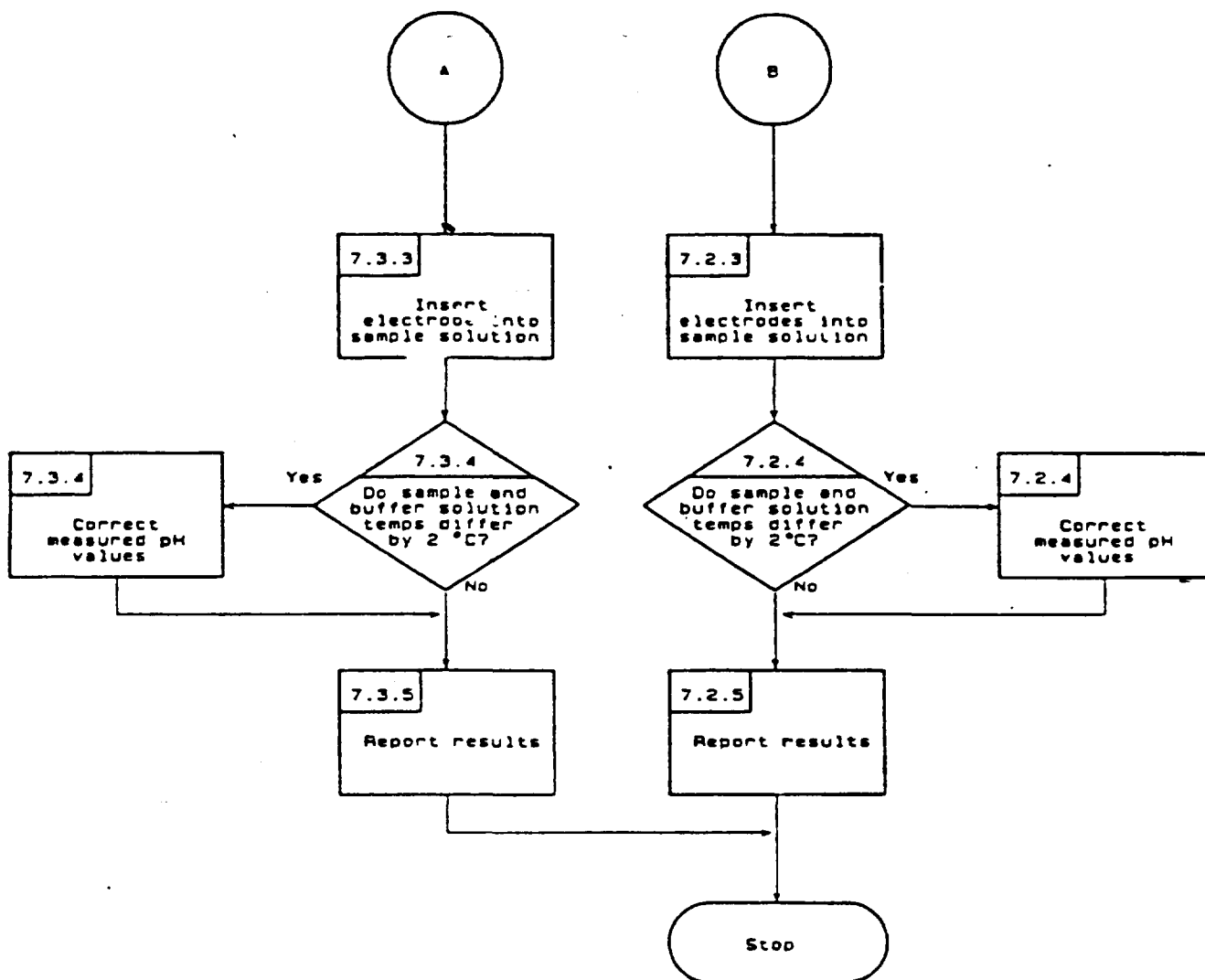
METHOD 9045

SOIL pH



METHOD 9045

SOIL pH
(Continued)



Appendix C

Sample Container Quality Control Certificates of Analysis

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Figure 6.1



Chemists in the Container Business™

I-CHEM RESEARCH

CERTIFICATE OF ANALYSIS

Analysis of Lot X0145023

June 18, 1990

Lot X0145023 has been cleaned to I-CHEM's Protocol A. Randomly selected samples from this lot were analyzed for pesticides by GC/Electron Capture Detector following EPA Method 608.

<u>Compound</u>	<u>Concentration</u> (ug/L)	<u>Compound</u>	<u>Concentration</u> (ug/L)
Aldrin	< 0.025	Dieldrin	< 0.05
Alpha-BHC	< 0.025	Endosulfan I	< 0.025
Beta-BHC	< 0.025	Endosulfan II	< 0.05
Delta-BHC	< 0.025	Endosulfan sulfate	< 0.05
Gamma-BHC	< 0.025	Endrin	< 0.05
4,4'-DDD	< 0.05	Endrin aldehyde	< 0.05
4,4'-DDE	< 0.05	Heptachlor	< 0.025
4,4'-DDT	< 0.05	Heptachlor epoxide	< 0.025

Randomly selected samples from this lot were also analyzed for extractables by GC/Mass Spectroscopy following EPA Method 625.

<u>Compound</u>	<u>Concentration</u> (ug/L)	<u>Compound</u>	<u>Concentration</u> (ug/L)	<u>Compound</u>	<u>Concentration</u> (ug/L)
Acenaphthene	< 5	Acenaphthylene	< 5	Anthrone	< 5
Benzo(a)anthracene	< 5	Benzo(b)pyrene	< 5	Benzo(b)fluoranthene	< 5
Benzo(k)fluoranthene	< 5	Benzo(g,h,i)perylene	< 5	Benzoic Acid	< 20
Biphenyl alcohol	< 5	4-Bromophenyl-phenylether	< 5	Biphenylphenol	< 5
Di-n-butylphthalate	< 5	4-Chloroaniline	< 5	4-Chloro-3-methylphenol	< 5
bis-(2-Chloroethoxy)benzene	< 5	bis-(2-Chloroethyl) ether	< 5	Azobenzene	< 5
2-Chloronaphthalene	< 5	2-Chlorophenol	< 5	4-Chlorophenyl-phenylether	< 5
Chrysene	< 5	Dibenz(a,h)anthracene	< 5	Dibenzofuran	< 5
1,4-Dichlorobenzene	< 5	3,3'-Dichlorobenzidine	< 5	2,4-Dichlorophenol	< 5
Dichlorophthalate	< 5	2,4-Dichlorophenol	< 5	Dichlorophthalate	< 5
4,6-Dinitro-2-methylphenol	< 20	2,4-Dinitrophenol	< 20	2,4-Dinitrotoluene	< 5
2,6-Dinitrotoluene	< 5	bis-(2-Ethylhexyl)phthalate	< 5	Fluoranthene	< 5
Fluorene	< 5	Hexachlorobenzene	< 5	Hexachlorobutadiene	< 5
Hexachlorocyclopentadiene	< 5	Hexachlorocyclopentadiene	< 5	Indene(1,2,3-cd)pyrene	< 5
Isophthalene	< 5	2-Methylnaphthalene	< 5	2-Naphthol	< 5
4-Naphthol	< 5	Naphthalene	< 5	2-Nitroaniline	< 20
3-Nitroaniline	< 20	4-Nitroaniline	< 20	Nitrobenzene	< 5
2-Nitrophenol	< 5	4-Nitrophenol	< 20	4-Nitroethylphenylamine	< 5
4-Nitro-di-n-propylamine	< 5	Di-n-octylphthalate	< 5	Perchlorophenol	< 20
Phenanthrene	< 5	Phenol	< 5	Pyrene	< 5
1,2,4-Trichlorobenzene	< 5	2,4,5-Trichlorophenol	< 20	2,4,6-Trichlorophenol	< 5
1,2-Dichlorobenzene	< 5	1,3-Dichlorobenzene	< 5	bis-(2-chloro-3-propyl) ether	< 5
4-nitroethyldichloroamine	< 5				

Contact our Technical Service Department if additional information is required.

Please keep this certificate for your records and to facilitate any necessary correspondence regarding this lot.

Randy E. Benson
Laboratory Manager
Hayward, California 94545 (415) 782-3905

New Castle, Delaware 19720 (302) 322-3808

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Figure 6.2



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I-CHEM RESEARCH

CERTIFICATE OF ANALYSIS

Analysis of Lot X0081023

April 13, 1990

Lot X0081023 has been cleaned to I-CHEM's Protocol A. Randomly selected samples from this lot were analyzed using ICP/Mass Spectroscopy, ICP/Atomic Emission Spectroscopy, Furnace Atomic Absorption, and Cold Vapor Atomic Absorption. The following analytical results were obtained.

Element	Concentration (ug/L)	Element	Concentration (ug/L)	Element	Concentration (ug/L)
Aluminum	< 20	Antimony	< 5	Arsenic	< 5
Barium	< 20	Beryllium	< 0.5	Cadmium	< 1
Chromium	< 10	Cobalt	< 10	Copper	< 10
Iron	< 30	Lead	< 2	Manganese	< 10
Mercury	< 0.2	Nickel	< 20	Selenium	< 2
Silver	< 5	Thallium	< 5	Vanadium	< 10
Zinc	< 10				

Selected samples from this lot were also analyzed for semivolatiles by GC/Mass Spectroscopy. The following analytical results were obtained.

Compound	Concentration (ug/L)	Compound	Concentration (ug/L)	Compound	Concentration (ug/L)
Acenaphthene	< 5	Acenaphthylene	< 5	Anthracene	< 5
Benz(a)anthracene	< 5	Benz(a)pyrene	< 5	Benz(b)fluoranthene	< 5
Benz(k)fluoranthene	< 5	Benz(g,h,i)perylene	< 5	Benzoic Acid	< 20
Benzyl alcohol	< 5	4-Bromophenyl-phenylether	< 5	Butylbenzylphthalate	< 5
Di-n-butylphthalate	< 5	4-Chloroaniline	< 5	4-Chloro-3-methylphenol	< 5
Bis-(2-Chlorophenyl)ether	< 5	Bis-(2-Chlorophenyl)ether	< 5	Azobenzene	< 5
2-Chlorophenol	< 5	2-Chlorophenol	< 5	4-Chlorophenyl-phenylether	< 5
Chrysene	< 5	9-Fluorene	< 5	Dibenzofuran	< 5
1,4-Dichlorobenzene	< 5	3,3'-Dichlorobenzidine	< 5	2,4-Dichlorophenol	< 5
Dibenzylphthalate	< 5	2,4-Dichlorophenol	< 5	Dibenzylphthalate	< 5
4,6-Dinitro-2-methylphenol	< 20	2,4-Dinitrophenol	< 20	2,4-Dinitrochlorobenzene	< 5
2,6-Dinitrochlorobenzene	< 5	Bis-(2-Ethylhexyl)phthalate	< 5	Fluoranthene	< 5
Fluorene	< 5	Hexachlorobenzene	< 5	Hexachlorobutadiene	< 5
Hexachlorocyclopentadiene	< 5	Hexachloroethane	< 5	Indeno(1,2,3-cd)pyrene	< 5
Isophthalene	< 5	2-Methylnaphthalene	< 5	2-Methylphenol	< 5
4-Methylphenol	< 5	Naphthalene	< 5	2-Nitroaniline	< 20
3-Nitroaniline	< 20	4-Nitroaniline	< 20	Nitrobenzene	< 5
2-Nitrophenol	< 5	4-Nitrophenol	< 20	2-Nitrophenylamine	< 5
2-Nitro-4-nitrophenylamine	< 5	Di-n-octylphthalate	< 5	Perfluorophenol	< 20
Phenanthrene	< 5	Phenol	< 5	Pyrene	< 5
1,2,4-Trichlorobenzene	< 5	2,4,5-Trichlorophenol	< 20	2,4,6-Trichlorophenol	< 5
1,2-Dichlorobenzene	< 5	1,3-Dichlorobenzene	< 5	Bis(2-chlorophenyl)ether	< 5
2-Nitro-4-nitrophenylamine	< 5				

Contact our Technical Service Department if additional information is required.

Please keep this certificate for your records and to facilitate any necessary correspondence regarding lot # X0081023

Randy E. Benson
Laboratory Manager

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Figure 6.3



Chemists in the Container Business™

I-CHEM RESEARCH

CERTIFICATE OF ANALYSIS

Analysis of Lot X0247013

September 18, 1990

Lot X0247013 has been cleaned to I-CHEM's Protocol A. Randomly selected samples from this lot were analyzed using ICP/Mass Spectroscopy, ICP/Atomic Emission Spectroscopy, Furnace Atomic Absorption, Flame Atomic Absorption, and Cold Vapor Atomic Absorption. The following analytical results were obtained.

Element/Compound	Concentration (ug/L)	Element/Compound	Concentration (ug/L)	Element/Compound	Concentration (ug/L)
Aluminum	< 80	Antimony	< 5	Arsenic	< 5
Barium	< 20	Beryllium	< 0.5	Cadmium	< 1
Calcium	< 500	Chromium	< 10	Cobalt	< 10
Copper	< 10	Iron	< 50	Lead	< 2
Magnesium	< 100	Manganese	< 10	Mercury	< 0.2
Nickel	< 20	Potassium	< 1000	Selenium	< 2
Silver	< 5	Sodium	< 5000	Thallium	< 5
Vanadium	< 10	Zinc	< 10	Cyanide	< 10

Selected samples from this lot were also analyzed for semivolatiles by GC/Mass Spectroscopy and pesticides by GC/Electron Capture Detector. The following analytical results were obtained.

Compound	Concentration (ug/L)	Compound	Concentration (ug/L)	Compound	Concentration (ug/L)
Acequinphos	< 5	Acequinphos	< 5	Anthracene	< 5
Benz(a)anthracene	< 5	Benz(a)pyrene	< 5	Benz(b)fluoranthene	< 5
Benz(k)fluoranthene	< 5	Benz(g,h,i)perylene	< 5	Benzoic Acid	< 20
Benzyl alcohol	< 5	4-Bromophenyl-phenyl ether	< 5	Butyl benzylphthalate	< 5
Di-n-butylphthalate	< 5	4-Chloroaniline	< 5	4-Chloro-3-methylphenol	< 5
bis-(2-Chloroethoxy)ethane	< 5	bis-(2-Chloroethyl) ether	< 5	Acetophenone	< 5
2-Chlorophenol	< 5	2-Chlorophenol	< 5	4-Chlorophenyl-phenyl ether	< 5
Chrysene	< 5	Bis(a,b,h)anthracene	< 5	Bis(a,b)anthracene	< 5
1,4-Dichlorobenzene	< 5	3,3'-Dichlorobenzidine	< 5	2,4-Dichlorophenol	< 5
Dichlorophthalate	< 5	2,4-Dichlorophenol	< 5	Dichlorophthalate	< 5
4,6-Dinitro-2-methylphenol	< 20	2,4-Dinitrophenol	< 20	2,4-Dinitrochlorobenzene	< 5
2,6-Dinitrochlorobenzene	< 5	bis-(2-Ethylhexyl)phthalate	< 5	Fluorenone	< 5
Fluorene	< 5	Hexachlorobenzene	< 5	Hexachlorobutadiene	< 5
Hexachlorocyclopentadiene	< 5	Hexachloroethane	< 5	Indeno(1,2,3-cd)pyrene	< 5
Isophthalate	< 5	2-Methylnaphthalene	< 5	2-Naphthol	< 5
4-Naphthol	< 5	Naphthalene	< 5	2-Nitroaniline	< 20
3-Nitroaniline	< 20	4-Nitroaniline	< 20	Nitrobenzene	< 5
2-Nitrophenol	< 5	4-Nitrophenol	< 20	0-Nitroethylphenylamine	< 5
0-Nitroethylphenylamine	< 5	Di-n-octylphthalate	< 5	Pentachlorophenol	< 20
Phenanthrene	< 5	Phenol	< 5	Pyrene	< 5
1,2,4-Trichlorobenzene	< 5	2,4,5-Trichlorophenol	< 20	2,4,6-Trichlorophenol	< 5
1,2-Dichlorobenzene	< 5	1,3-Dichlorobenzene	< 5	Bis(2-chloroisopropyl) ether	< 5
0-Nitroethylphenylamine	< 5	Aldrin	< 0.025	4,4'-DDE	< 0.05
Endosulfan II	< 0.05	Alpha-BHC	< 0.025	4,4'-DDD	< 0.05
Endosulfan sulfate	< 0.05	Beta-BHC	< 0.025	4,4'-DDT	< 0.05
Endrin	< 0.05	Gamma-BHC	< 0.025	Endrin	< 0.05
Endrin Aldehyde	< 0.05	Endrin-BHC	< 0.025	Endosulfan I	< 0.025
Endosulfan	< 0.025	Endosulfan epoxide	< 0.025		

Contact our Technical Service Department if additional information is required.

Please keep this certificate for your records and to facilitate any necessary correspondence regarding this lot.

Randy E. Benson
Laboratory Manager
Hayward, California 94545 (415) 782-3905

New Castle, Delaware 19720 (302) 322-3808

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Figure 6.4



Chemists in the Container Business™

I-CHEM RESEARCH

CERTIFICATE OF ANALYSIS

Analysis of Lot 0207023

August 03, 1990

Lot 0207023 has been cleaned to I-CHEM's Protocol C. Randomly selected samples from this lot were analyzed using ICP/Mass Spectroscopy, ICP/Atomic Emission Spectroscopy, Furnace Atomic Absorption, Flame Atomic Absorption, and Cold Vapor Atomic Absorption. The following analytical results were obtained.

<u>Element</u>	<u>Concentration</u> (ug/L)
Aluminum	< 80
Antimony	< 5
Arsenic	< 5
Barium	< 20
Beryllium	< 0.5
Calcium	< 100
Cadmium	< 1
Chromium	< 10
Cobalt	< 10
Copper	< 10
Iron	< 50
Lead	< 2
Magnesium	< 100
Manganese	< 10
Mercury	< 0.2
Nickel	< 20
Potassium	< 100
Selenium	< 2
Silver	< 5
Sodium	< 100
Thallium	< 5
Vanadium	< 10
Zinc	< 10

Contact our Technical Service Department if additional information is required.

Please keep this certificate for your records and to facilitate any necessary correspondence regarding this lot.

Randy E. Benson
Laboratory Manager

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Figure 6.5



I-CHEM RESEARCH

CERTIFICATE OF ANALYSIS

Analysis of Lot 0114033

April 26, 1990

Lot 0114033 has been cleaned to I-CHEM's Protocol C. Randomly selected samples from this lot were analyzed using ICP/Mass Spectroscopy, ICP/Atomic Emission Spectroscopy, Furnace Atomic Absorption, Flame Atomic Absorption, and Cold Vapor Atomic Absorption. The following analytical results were obtained.

<u>Element</u>	<u>Concentration</u> (ug/L)
Aluminum	< 80
Antimony	< 5
Arsenic	< 5
Barium	< 20
Beryllium	< 0.5
Calcium	< 100
Cadmium	< 1
Chromium	< 10
Cobalt	< 10
Copper	< 10
Iron	< 50
Lead	< 2
Magnesium	< 100
Manganese	< 10
Mercury	< 0.2
Nickel	< 20
Potassium	< 100
Selenium	< 2
Silver	< 5
Sodium	< 100
Thallium	< 5
Vanadium	< 10
Zinc	< 10

Contact our Technical Service Department if additional information is required.

Please keep this certificate for your records and to facilitate any necessary correspondence regarding lot # 0114033.

Randy E. Benson
Laboratory Manager

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Figure 6.6



I-CHEM RESEARCH

Chemists in the Container Business™

CERTIFICATE OF ANALYSIS

Analysis of Lot Number W9163013

The following compounds were analyzed for but not detected, or detected at a concentration of less than 5 micrograms/liter:

Chloromethane	Bromomethane	Vinyl Chloride
Chloroethane	Methylene Chloride	Acetone
Carbon Disulfide	1,1-Dichloroethane	1,1-Dichloroethene
Chloroform	1,2-Dichloroethane	trans-1,2-Dichloroethene
2-Butanone	1,1,1-Trichloroethane	Carbon tetrachloride
Vinyl acetate	Bromodichloromethane	1,2-Dichloropropane
Trichloroethene	Dibromochloromethane	1,1,2-Trichloroethane
Benzene	Bromoform	cis-1,1-Dichloromethane
2-Hexanone	4-methyl-2-pentanone	Tetrachloroethene
Toluene	Chlorobenzene	Ethylbenzene
Styrene	Aylenes	1,2-Dichlorobenzene
1,3-Dichlorobenzene	1,4-Dichlorobenzene	trans-1,2-Dichlorobenzene
1,1,2,2-Tetrachloroethane	2-Chloroethyl vinyl ether	

Please retain for your records and to aid in any correspondence with I-CHEM regarding lot W9163013.

Certified
Anita Rudd
President

Section No. 6
Revision No. 1
Date: October 1990
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Figure 6.7



10-9-90

CERTIFICATE OF ANALYSIS

Analysis of Lot Number EI 0601

The following compounds were analysed according to EPA Method 502.2 criteria and not detected, or detected at a concentration of less than 1 microgram per litre:

Dichlorodifluoromethane	1,2-Dibromoethane
Chloromethane	Chlorobenzene
Vinyl Chloride	Ethylbenzene
Bromomethane	1,1,1,2-Tetrachloroethane
Chloroethane	m-Xylene
Trichlorofluoromethane	p-Xylene
1,1-Dichloroethene	o-Xylene
Methylene Chloride	Styrene
trans-1,2-Dichloroethene	Isopropylbenzene
1,1-Dichloroethane	Bromoform
2,2-Dichloropropane	1,1,2,2-Tetrachloroethane
cis-1,2-Dichloroethene	1,2,3-Trichloropropane
Chloroform	n-Propylbenzene
Bromochloromethane	Bromobenzene
1,1,1-Trichloroethane	1,3,5-Trimethylbenzene
1,1-Dichloropropene	2-Chlorotoluene
Carbon Tetrachloride	4-Chlorotoluene
Benzene	tert-Butylbenzene
1,2-Dichloroethane	1,2,4-Trimethylbenzene
Trichloroethene	sec-Butylbenzene
1,2-Dichloropropane	p-Isopropyltoluene
Bromodichloromethane	1,3-Dichlorobenzene
Dibromomethane	1,4-Dichlorobenzene
cis-1,3-Dichloropropene	n-Butylbenzene
Toluene	1,2-Dichlorobenzene
trans-1,3-Dichloropropene	1,2-Dibromo-3-Chloropropane
1,1,2-Trichloroethane	1,4-Trichlorobenzene
1,3-Dichloropropane	Hexachlorobutadiene
Tetrachloroethene	Naphtalene
Dibromochloromethane	1,2,3-Trichlorobenzene

Please retain this certificate of analysis for your records and to aid in any correspondence with Macro Scientific, Inc. regarding Lot Number EI 0601

MACRO
SCIENTIFIC, INC.

4805 FRANCE AVE N
MINNEAPOLIS, MINNESOTA 55412
PHONE (612) 536-1234
FAX (612) 536-1051